RESEARCHES ON WATER RESISTANT GLUES

BY

S. BUTTERMAN

ARMOUR INSTITUTE OF TECHNOLOGY
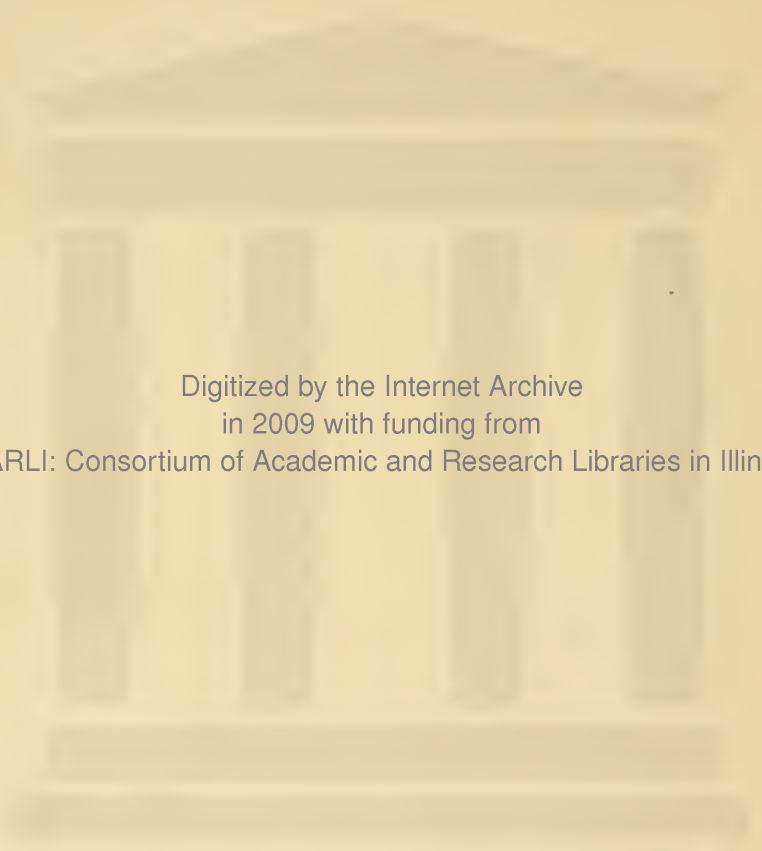
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Researches On Water Resistant Glues

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Samuel Butterman

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APPROVED:

Harry M. McCormack
Professor of Chemical Engineering

H. M. Raymond
Dean of Engineering Studies

Dean of Cultural Studies

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INTRODUCTION.

Necessity for Water Resistant Glues

Coincident with our entry into the Great War it was rather abruptly realized that an urgent necessity for water-resistant glues for use in aircraft construction existed. In this country relatively little was known at that time regarding the preparation of such a product. During the period of our participation in the Struggle the high price commanded by such a glue stimulated an active competition which has created several satisfactory water-resistant glues that are at present on the market.

The need for glues of this type becomes manifest when it is considered that aircraft parts experience considerable variation in moisture content during the course of flight, by reason of direct contact with clouds, and

because of the precipitation of moisture upon
chilled parts of machines descending from high
elevations into the warm air-strata of lower
altitudes. Furthermore, before a flight, the
air trapped within wing surfaces and confined
by wing covering has a relative humidity cor-
responding to that which exists at the level
of the earth; on rising into higher elevations
the low temperatures encountered cause satur-
ation of this air to be exceeded and moisture
is accordingly deposited upon the wing struc-
tures. Again, marked changes in humidity are
encountered during storage in the damp holds
of vessels transporting planes overseas, and
also because of climatic changes. Hence, if
danger from loose joints is to be reduced to
a minimum, the need for water resistant glues
to replace the standard grades of animal
glue is obviously urgent in connection with
aircraft production.

Application to Aircraft Structures

Plywood Construction.--From the fact that
every effort is made to decrease the weight
of aircraft as far as practical, we find the
most predominant structural material employed
is wood. In certain elements of construction
(wing beams, struts, etc.,) the wood is used
in the form of either solid or laminated
stock, but where very light structures are
necessary, a relatively high development of
strength may be obtained only by resorting
to plywood construction.

Plywood construction involves the use of
a simple, ingenious method of improving upon
Nature's method of producing wood. If a
thin slab of wood is cut from a tree parallel
to the fiber length (not across it), this slab
will be found to yield to bending more readi-
ly about its vertical axis than its horizon-
tal axis, due to the resistance which the

fiber offers to deformation. Hence the thin slab has a negligible amount of stiffness in one direction, and, accordingly for practical purposes has no strength. To overcome this shortcoming the thickness must be increased - which means increased weight.

Plywood construction avoids this difficulty and meets the necessity of lightness by merely combining the thin slab of wood with two others located on either side of it but arranged in such a manner that the direction of grain in the outside plies is perpendicular to that in the central slab. It is this reversal of grain which constitutes the improvement upon Nature inasmuch as the artifice provides resistance to deformation regardless of the direction of the acting force and so creates a member which, for the same weight, is many times stiffer and stronger than solid stock of the same species.

This built up member is called a veneer panel or sometimes plywood; the individual plies are veneers. The outer plies are faces and the central one is designated a core. Where the plywood is built up of more than three plies the central plies are termed cross-bands or cross-banding.

Points of Application of Glue.--The points of application of glue in the airplane are becoming more numerous. The plywood which forms the entire skim of fuselage construction, in most machines, is joined exclusively with some form of water-resistant glue. The use of plywood has also been extended to the manufacture of cowlings, engine bed-plates, aviator seats, wing-rib webs, etc. In the most improved design struts and beams of irregular hollow-section have been devised, in order to reduce the weight of the member per unit of strength;

this necessitates laminated construction
and hence involves the use of glue. Fur-
thermore with the aid of water-resistant
glue it has become safer to utilize short
lengths of stock (heretofore discarded as
waste) by resorting to built up structures
composed of short lengths combined through
lap joints. In the case of propeller man-
ufacture which requires use of the most ex-
pensive species of wood (viz; walnut, ma-
hogany, oak, cherry, etc.) production of
scrap has been reduced to a minimum by us-
ing laminated construction; the rough lam-
inations each differ in shape and length to
such an extent as to approximate, when as-
sembled and glued,the peculiar contour of
the finished propeller.

Such practices as these become more
practical with the use of water-resistant
glues of good quality and are valuable in-

asmuch as they not only reduce initial
cost but they effect a conservation of
material and so reduce to a minimum the
danger of depletion of limited sources
of species.

THE PROBLEM

Need for Development of Glue-Formu-
lae.--Early in the period of our parti-
cipation in the Great War the responsi-
bility for the development of our air-
craft program rested with the Signal
Corps of the Army. This organization
upon becoming aware of the necessity of
water-resistant glue for aircraft pur-
poses soon discovered that but one com-
pany in the country was manufacturing
such a product and this - according to
a secret process. Having possession of
a secret glue formula by which to manu-
facture, this company was in a position

to dictate prices and monopolize the
field.   Other companies were prevented
from contributing to the aircraft pro-
gram because no glue formulae were a-
vailable for general use. Since the
one company was unable to supply suf-
ficient glue to insure the yearly pro-
duction of plywood required for the
aircraft program, the Signal Corps, in-
order to insure sufficient production,
found itself under the necessity of
securing the co-operation of the Di-
vision of Forestry, Department of Ag-
riculture which undertook a study to
determine methods of preparing water-
resistant glues.

It was anticipated that the ad-
vantages which would accrue from the
development of a high grade glue for-
mula would be several: first, if made

available for general use it would insure
an adequate contribution of plywood to the
aircraft program; if withheld from general
dissemination, mere knowledge of its de-
velopment should stimulate effort on the
part of companies, which had recently en-
tered the field, to improve their product
in order to survive competition; thus the
tendency would be towards the manufacture
of higher grade plywood; furthermore, pos-
session of such a formula would enable the
Signal Corps to support stringent specifi-
cations (based on the capabilities of the
new formula) covering qualities of ply-
wood acceptable for aircraft purposes.
Such procedure would likewise serve to
promote improvement in grade of commercial
plywood.

Statement of the Problem.--The prob-
lem then, as encountered by the writer,

was to develop a formula for a water-re-
sistant glue of good quality. The research
was to be undertaken at the Forest Products
Laboratory at Madison, Wisconsin, in co-
operation with the University of Wisconsin.

SOLUTION OF THE PROBLEM

Section I - Survey of Available
Information

Types of Water-Resistant Glue Available

Two types of water-resistant glue
seemed already to be well recognized; one
based on soluble blood albumin and the
other on casein.

Blood Albumin Glue.--Blood albumin, a
product derived from the blood collected
in the killing beds of slaughter houses,
when dissolved in certain alkaline solu-
tions yields a glue of high water-resis-
tant character upon coagulation of this

solution by heat. This quality is due to
the fact that the coagulum formed is insol-
uble and this behavior constitutes the prin-
ciple of all glues of the blood albumin
type. The disadvantages of this type of
glue are that expensive hot plate equip-
ment is required, and special means must
be taken to coagulate the glue when heavy
stock is glued.

Casein Glue. Closely approaching blood
albumin glue in the property of resisting
the deteriorating influence of water is the
type known as casein glue. Casein itself
is the white solid which forms during the
familiar souring of milk. The casein,
according to available information, could
be prepared in the form of glue by dis-
solving it in milk of lime. A glue so pre-
pared is strictly a "cold" glue and has the
extreme advantage that joints made with it

need not be gotten under pressure quickly, and requires merely the simplest kind of pressing equipment.

Casein - Occurrence, Manufacture, Properties.

Inasmuch as it was desired to develope a glue with casein as a base, it now became necessary to undertake a detailed study of the nature and applications of casein.

Occurrence. Casein occurs as the principal proteid of milk. As a general thing the milk of mammals is an opaque, whitish or faintly yellow liquid (often amphoteric to litmus) consisting of water, fat, casein, albumin, milk sugar (lactose) and inorganic constituents; the most important constituents are the fat and the casein. Cow's milk is the chief variety and shows the following average analysis:

Fat	-	3.4	%
Casein	-	3.2	"
Albumin	-	0.6	"
Lacto-protein	-	0.1	"
Lactose	-	4.5	"
Ash	-	0.7	"
Water	-	87.5	"

Free casein is not dissolved in milk, but occurs associated with calcium in the form of a suspensoid, as can be proved by filtering milk through a porous earthenware plate; the filtrate obtained consists merely of a solution of milk sugar and saline constituents; the fat and combined casein remain on the filter. The combined casein can also be thrown out unchanged by high centrifugal action, demonstrating again the suspensoid state of its existence.

The exact nature of this union of

casein with calcium, in the form in which
it occurs in milk, has been the subject
of much controversy. Some authorities main-
tain that the two ingredients are present in
stoichiometrical proportions and exist as a
true compound; others hold that the calcium
is adsorbed by the casein and the union is
an adsorption compound. The truth is still
a matter of question. The compound is gen-
erally designated as calcium - caseinate.

   Manufacture.--When fresh, whole milk is
left at rest, or is set in very rapid, ro-
tary motion by means of a centrifuge, the
cream, or portion richest in fat, collects
on the surface, and if this be skimmed off,
skim milk remains. This latter is the raw
material for the preparation of the second
chief product of milk, viz: casein.

   Casein is obtained from skim milk by liber-
ating it from its combination with calcium

and taking advantage of the insolubility
of the product which is base-free casein
or simply - casein. This liberation is
effected in one of two ways, viz:

I. Displacement of the calcium by
acid using,

Lactic acid

Hydrochloric acid

Sulphuric acid

Acetic acid

etc.

II.Coagulation by the chemical fer-
ment rennet.

Natural Sour Method. This method of
liberating free casein from the combined
form in which it exists in milk depends
upon the fact that milk, even when fresh-
ly obtained from the cow, contains lactic
acid bacteria. These bacteria feed upon
the sugar (lactose) of milk and one of the

products of their life phenomena (fermentation) is lactic acid. The concentration of lactic acid increases progressively and after some time has become high enough to liberate all of the casein. This is the familiar souring of milk.

The exact nature of the mechanism by which the acid causes the calcium to split off from its combination with casein, whether by simple chemical double decomposition or through adsorption phenomena remains a matter of controversy.

Mineral Acid Method.--The natural sour method of liberating casein requires considerable time because the development of acid is so gradual that a number of hours must elapse before a sufficient concentration is reached to complete the precipitation. For example it is not uncommon creamery practice to allow the process to go on over-night. Commercially the time is mater-

ially shortened by introducing, as a sub-
stitute for lactic acid, some mineral acid
which is commercially available, such as
hydrochloric or sulphuric acids. The acid
is introduced in sufficient quantity to
raise the concentration to the desired
point practically at once. Hence the en-
tire process can be accomplished in about
one hour.

Various other acids such as acetic
acid, etc., are sometimes used.

Rennet Coagulation.--Rennet casein is
obtained by treating skim milk with rennet,
a chemical ferment obtained from the fourth
stomach of a suckling calf. The rennet pro-
cess differs from the acid methods in that
chemical precipitation does not occur; the
action is one of simple coagulation; the
solution becomes neither acid nor alkaline.

The result of the action of rennet in

coagulating milk is wholly unlike the effect
produced by acids, from a chemical view point.
The curd formed by this action is not the
simple base-free casein, but the original
calcium caseinate itself with a certain strik-
ing modification; this product is generally
called calcium para-caseinate.

Properties. Casein is a very complex
chemical compound belonging to the general
class of proteins. It has the following
percentage composition:[1]

C 52.96 %
H 7.04 " to 7.53 %
N 15.6 " to 15.91 "
O 22.78 "
S 0.75 " to 0.82 "
P 0.8 " to 0.84 "

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[1] Thorpe - Dictionary of Applied Chemistry

From its intimate union with calcium in milk, we find commercial caseins associated with varying quantities of calcium, generally recorded as ash content. The ash content of acid-prepared caseins is given as ranging from 1 to 3 per cent; rennet casein is notoriously higher in ash content ranging from 7 to 8 per cent.

Casein, when pure, is a white amorphous body insoluble in distilled water or very dilute salt solutions. It is soluble in 5 per cent salt solution and in hot 50 per cent alcohol. It is odorless and forms when dry a crumbling, horny mass. It is an amphoteric substance exhibiting the typical dual combining capacities characteristic of proteins.

The basic properties of casein have not been so extensively studied. It dis-

solves easily in moderately dilute acids,
more easily at higher temperatures, forming sol-
uble compounds which are either combinations
of acid with the protein or decomposition pro-
ducts, depending on conditions. Some hold
that simple addition products of the casein
and acid are formed, such as casein hydro-
chloride. These casein salts are very un-
stable and decompose on dialysis with sep-
aration of free casein; they are precipita-
ted from solution by addition of just enough
alkali to neutralize the combined acid; treat-
ment with such salts as sodium or calcium
chloride, sodium sulphate, etc. accomplish
the same effect.

If a "weak" acid such as acetic acid
be employed for precipitation, the acidity
of the mixture is insufficient (because of
limited dissociation) to transform the ca-
sein into a base and a considerable excess

of acetic acid may be added without danger of loss of material through re-solution of the protein. Accordingly for quantitative estimation of casein the precipitant unvariably used is acetic acid. Concentrated acids char ("burn") casein and readily dissolve it.

The acid properties of casein have been more extensively investigated. Free casein suspended in water acts as an acid expelling carbon dioxide from carbonates and bi-carbonates forming a so called salt with the base; and in this way, according to Osborne, the ammonium, potassium, sodium, lithium, magnesium, strontium and calcium caseinates are prepared.

In the presence of alkalies casein likewise exhibits its acid character, uniting with fixed alkalies, ammonia, alkaline carbonates and phosphates, borax,

sodium silicate, etc., passing into various states of solution; those with alkalies are nearly transparent and exhibit the least viscosity; the compounds with the alkaline earths give solutions which are opalescent or milky in appearance and of high viscosity. On heating with alkali decomposition occurs, ammonia being evolved; as the hydrolysis progresses the liquid becomes extremely thin. The alkali "salts" of casein, but not those of the alkaline earths, diffuse through porous earthenware.

The so-called caseinates formed above are precipitated, from their solutions, very easily by the addition of dilute acid, (example curdling of milk), the precipitate redissolving in excess of acid. They are also thrown out of solution by sodium chloride and sulphates of magnesium sodium, ammonium, zinc, copper, aluminum and other salts.

Rennet casein or para-casein is identical in properties with free casein with the single exception that it is insoluble in the presence of soluble calcium salts.

Applications. For many years the sole use of casein was as a foodstuff, and, it is only through the scientific researches of the past few decades into the albuminoid substances that attempts have been made to utilize this body technically. At present casein finds an extremely wide range of applications and its uses are becoming more manifold daily. It is used in pastes, glues, card-board manufacture, paper sizing and glazing, leather dressing, soap manufacture, cotton sizing, dressing for textiles, finishing, waterproofing of fabrics, calico printing, cask glazing, artificial bone and ivory, billiard balls, tortoise shells, amber, ebony, picture mouldings, sealing for bottles,

cabinet and pianoforte, making emulsions, fining wines, boot polishes, polishes, films, etc. Furthermore, its original application as a foodstuff has not been lost sight of; its high nutritive value to man and its ready assimilability have led to the preparation of number of artificial foods, viz: Sanotogen, Nutrose, etc.

Casein as a Glue Base.-The employment of casein as a glue base is of fairly ancient date; its property of dissolving in alkaline liquids to form a good mucilage was known to the ancients. Yet it is only in the last thirty years that it has been placed on the markets of Europe under the name "cold" glue. It has not yet made much progress notwithstanding the advantage that such a glue is inodorous, ready for immediate use and requires no heating.

The literature on the subject of casein

glue contains many recipes and formulae.
In general the ingredients recommended are
the alkaline earths, their salts, ammonia,
etc. Formulae for water-resistant glues
invariably recommend lime as the solvent,
along with certain other additional in-
gredients to confer desirable properties,
such as alkali carbonates, phosphates, so-
dium silicate, borax, arsenates, arsenites
and an endless number of other ingredients
to confer certain desirable properties.
Formaldehyde is recommended to increase
water-resistance qualities, sodium arsen-
ate and caustic soda to improve adhesive
strength, etc.

Section II - Preliminary
Experimentation
Early Experiments and Observations

For the purpose of acquiring famil-
iarity with the behavior of casein under

the influence of the so-called "solvents"
described in the foregoing text, a great
number of experiments were performed us-
ing various proportions and combinations
of the solvents with a suspension of cas-
ein in water. It became manifest early
that solution of the casein particles,
was most rapid if a casein of fine mesh
were employed, and, if the latter were
allowed to soak for some time in water
before mixing with the solvent. Since
it was well recognized that promising re-
sults could be obtained from a glue pre-
pared from lime and casein this simple
combination served as the starting point.

For this work there was available a
number of technical caseins varying in
quality from practically colorless, sweet
smelling products through all gradations
of color from light yellow to dark brown

bodies; some putrid from the rancidity
of their high fat contents; others ex-
hibiting sour and even highly offensive
odors depending upon the degree of pu-
trefaction developed as a result of
careless manufacture. It was apparent
at once that commercial caseins are far
from uniform in quality.

The caseins, sifted to a fineness
of 20 to 40 mesh were soaked in a quan-
tity of water; after some time the par-
ticles were observed to swell and absorb
the free water producing a mush consis-
tency which is hereafter designated as
the "soak". Various proportions of milk
of lime of different strengths were now
stirred in and the agitation was continued
until a uniform texture, practically free
from particles, was obtained.

Sufficient water was added to produce

a ready flowing consistency. Under the
influence of the lime (solvent) and pro-
per agitation it was observed that the
casein gradually disappeared into the
body of the fluid, the latter develop-
ing at the same time a gradual increase
in viscosity, a milky opalescent appear-
ance, and marked mucilaginous qualities.
These preparations invariably exhibited
a rapid, spontaneous increase in viscos-
ity with subsequent loss of fluidity and
transformation into a rigid rubbery mass
of no utility for the purposes under con-
sideration. In general the range of use-
fulness (period during which preparation
retained its fluid qualities-designated
hereafter as "life") was exceeded in a
period short of one hour.

It was further observed that the glue-
life decreased in proportion as the lime

content was increased, notwithstanding
the fact that repeated additions of lime
invariably reduced the viscosity progres-
sively and gave higher degrees of fluidity.
Yet the minimum quantity of lime which ser-
ved to cause proper solution of the casein
did not produce an increase in life suffi-
cient to prolong it beyond the extent stat-
ed above, without addition of such exces-
sive quantities of water as to destroy most
of the adhesive qualities of the mixture.

This very limited life was admittedly
a very serious shortcoming of the casein-
lime glues because in its commercial appli-
cation the glue would exceed its period of
usefulness in such a short time as to make
its use impractical.

A very striking feature, which pro-
voked much annoyance, as observed in the

foregoing experiments was the enormous
variation in viscosity developed by dif-
ferent caseins under the applications of
the same formula. One casein yielded with
a fixed quantity of water and lime a glue
of satisfactory working consistency where-
as the next casein tried might give a
thick, heavy mixture of no utility be-
cause of low degree of fluidity. While
this difficulty could be overcome by vary-
ing the quantity of water to meet the re-
quirement of the specific casein used, the
fact remained that technical caseins for
some reason, differ widely in their water-
absorbing capacities. This circumstance,
obviously, would not admit of the evolu-
tion of a standard formula with fixed pro-
portion of water; the latter, apparently,
had to be determined for each new casein
by some routine method of trial, unless

the cause underlying this variation were
determined, on the basis of which, means
for selecting caseins of a fixed water-
absorption capacity would become avail-
able. This characteristic divergence in
behavior of technical caseins so far ex-
perienced seemed to conform to the gener-
al impressions prevalent among commercial
consumers of casein. One manufacturer
will specify a preference for natural-
sour casein, whereas another maintains
that sulphuric acid casein only could
serve his process, etc. No one seemed to
have a conception of the reason for the
difference - yet all were agreed that the
behavior of technical caseins is far from
uniform.

Interpretations.--The striking be-
havior of the casein-lime mixtures, the
pronounced viscosity, opalescence, and

the rapid transformation of state from a
fluid mixture to a rigid mass, none of
which qualities are characteristic of true
solutions, is conclusive evidence that the
systems to be dealt with are emulsoid col-
loids of typically high viscosity, to which
they owe their adhesive qualities. Revert-
ing to the preliminary soaking of the cas-
ein, Robertson[1] points out that wet, freshly
precipitated casein dissolves rapidly in the
presence of solvents but the physical pro-
perties of dry, granular casein hinder so-
lution. Experience has shown that if the
casein particles are allowed to soak and
swell in water they yield readily to the
action of the lime, while if the dry gran-
ular casein were stirred up with lime and
water a lumpy mixture obtains.

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[1] Robertson, T.B.- Physical Chemistry of the
Proteids

This is entirely analagous to the fact
that if gelatine is thrown into water and
the mixture is immediately heated imper-
fect lumpy sols are likely to be obtained,
while, if the gelatine is first allowed
to soak and swell in water before heating,
the process of solation occurs without
trouble.

The literature reveals that casein
dissolves in caustic alkalies forming
transparent liquids which filter unchanged
through porous earthenware. The writer has
prepared such solutions with borax which
are of low viscosity and remain fluid with
no signs of setting for days. These give
indication of a condition of true solution.
In the case of the opalescent, highly vis-
cous alkaline earth "solutions" of casein
the situation is different. In the pres-
ence of calcium hydroxide, the casein, in

the language of colloid chemistry, prob-
ably adsorbs hydroxyl ions and dissolves.
As more lime is added it follows, from the
adsorption law, that more hydroxyl ions
should be adsorbed - the degree of dis-
persity should increase; that is the size
of particles should more nearly approach
that of molecular subdivision and, accord-
ingly, the viscosity should decrease. This
was actually observed in the case of lime
but is even more striking when caustic so-
da is used, small additions causing a ma-
terial decrease in viscosity.

Another influence now begins to mani-
fest itself - that of the powerful coagu-
lating tendency of the divalent kation
calcium. The system begins to exhibit its
inherent instability, the extremely minute
colloidal particles increase in size by
coalescence, the viscosity increases spon-

taneously, the mixture becomes sluggish and gelatinization sets in, i.e., it "sets". This coagulation is irreversible and to this circumstance is attributed the ability of the glue to resist solvent action of water. As the calcium ion concentration increases the coagulating action becomes more powerful and, as observed, gelation takes place more quickly.

As a resume of the observations of the foregoing experiments two features are dominant, first, the diverse character of technical caseins and secondly, the inherent instability of the simple case-lime combination. The work immediately following was promptly undertaken with a view to investigate the first and to overcome the second.

Preparation of Casein Samples

As an aid in determining the reason·
for the diversity in character of caseins,
it was proposed to carefully prepare a ser-
ies of caseins according to the various
commercial methods, with the idea of cor-
relating casein behavior with method of
manufacture, factor affecting the latter,
etc. The actual manufacture of the curd
was performed at the University of Wiscon-
sin dairy.

Caseins were prepared by the natural
sour method, by the mineral-acid methods
and by the rennet process. These were
dried upon trays in a current of warm air
and ground to flour in a pebble mill.

Actual examination of these caseins
for glue qualities was deferred until a
promising formula was developed as is
subsequently described.

Stabilization of Casein - Lime Mixtures

Before undertaking any tests for water-
resistance qualities of the casein-lime mix-
tures which had already demonstrated their
inherent instability, steps were now taken
to attempt to stabilize these mixtures in
order to make them sufficiently practical
to warrant further development. Inasmuch
as the literature revealed no method of pro-
cedure it was decided to study the behavior
of glues prepared according to the most pro-
mising of the many recipes available and to
experiment with the use of gum arabic and
gelatine, etc., because of their notorious
stabilizing influence on colloidal solutions.
To this end there was undertaken a prolonged
series of experiments in which the simple
casein-lime combination was incorporated
with various proportions and combinations of
borax, sodium carbonate, tri-sodium phos-

phate, caustic soda, ammonia, sodium sili-
cate, zinc oxide, gums, gelatine, etc., fol-
lowing a tedious routine to exhaust possi-
bilities.

As a result of the above work the very
striking discovery was made that water glass
exerts a material retarding influence on the
gelatinization of casein glue. The formula
used, giving ingredients and proportions, is
given in formula VIII - #5, below.

Formula VIII - #5

```
100 gr. casein) Soak
300 gr. water ) ½ hour)
     +                )
 25 gr. CaO   )          )Mix)
125 gr. water )          )   )
     +                       )Mix
 50 gr. sodium silicate  )
```

The casein was soaked in water for 1/2 hour
following which the milk of lime was stirred
in; after a few moments of stirring the wa-
ter-glass was poured in and the agitation
was continued until the mixture was free
from lumps. These proportions gave, with

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the casein used, a very watery fluid, which
remained fluid for approximately 30 hours.

The fact that a casein-lime mixture,
mixed practically as normally, had retained
its fluid character for many hours by vir-
tue of the addition of silicate, led to the
idea of determining whether the retarding
influence is dependent upon the stage of
mixing at which the water-glass is intro-
duced. It was first proposed to allow a
simple casein-lime glue to increase in
viscosity until a desirable working con-
sistency was reached at which point the
effect of introducing various amounts of
"silicate", in the hope of maintaining
this viscosity and fluidity for a pro-
longed time, would be studied. The next
series of experiments were for the pur-
pose of observing the effect of intro-
ducing various amounts of silicate im-

mediately after the lime had been stirred
in.

The data obtained from the first pro-
cedure are given below in Table I.

Table I - Effect on Life of Stage of Intro-

duction of Silicate

Proportions of Ingredients						
Casein	Water	:Milk of: Lime	Silicate			:Life
			:Amt.	:Time intro- :duced (after: lime		
gr.	gr.	gr. (1-5)	:gr.	min.		:hrs.
100	300	150	52	40		:2-1/2
100	300	150	25	60		: 3/4
100	300	150	60	60		:1-3/4
100	300	150	52	75		:1-1/2

The data from the second series of experi-
ments, in which was studied the effect of
increasing quantities of silicate introduced
immediately succeeding the addition of lime

are given in Table II. The formula employed
was the same as that used above save that
the silicate was varied as shown; the cor-
responding life is also shown.

Table II - Effect of amount of Silicate on

Life

Casein	:	Silicate	:	Life
gr.	:	gr.	:	hrs.
100	:	25	:	1-3/4
100	:	36	:	5
100	:	52	:	30
100	:	58	:	32

Analyzing the data from Table I and Table
II the following features are dominant:
1. Gelatinization of casein-lime mixtures
is delayed by the presence of silicate;
the extent of the retarding influence is
actuated by the stage of mixing at which
the silicate is introduced.
2. The retarding influence is effective

only if the silicate is added imme-
diately after the lime.

Mechanism of Stabilization.--
With regard to the explanation of the
retarding influence of silicate upon
the gelation of the casein-lime mix-
ture, two possibilities present them-
selves:

I. Emulsoid colloids, such as sil-
icate, have a tendency to "protect" less
stable sols. According to Bechhold's
idea of protection, the latter is the re-
sult of adsorption; a thin layer of the
silicate is adsorbed at the interface
and thus confers its stability on the ad-
sorbing particles of calcium caseinate.
The latter are thus effectively prevented
from forming aggregates until the relative-
ly stable silicate membrane gels - hence
gelation of the mixture is itself delayed

to this point.

II. Another possibility is that the
silicate maintains a very low concentra-
tion of calcium ions through formation
of insoluble calcium silicate. This cir-
cumstance, obviously would slow down co-
agulation.

It is of course possible that both
actions described may be in operation at
once.

Section III - Investigation of Glue
Qualities of Silicate Mixtures

In view of the promising aspect of
casein-lime-silicate mixtures, particu-
larly as combined in formula VIII - #5
(preceeding), it was next in order to
test the water-resistance and strength
qualities of this combination to deter-
mine the degree of its utility. For this
purpose the following series of routine

tests were to be applied.

Methods of Testing Glue Qualities

Obviously, rational tests for the determination of glue capabilities must be such as determine whether glued joints will effectively resist the deteriorating conditions encountered in service. Glued joints in aircraft service must contend with fresh water, lubricating oil, gasoline and, in the case of hydroplanes, with Salt-water. To study the relative severity of these conditions the following tests had already been completed on veneer-panel specimens.

1. Soaking in fresh water at room temperature for two weeks.

2. Soaking in salt water at room temperature for two weeks.

3. Boiling in fresh water for 24 hours followed by baking at 212°F. for 24

hours.

4. Boiling in salt water for 24 hours followed by baking at 212°F for 24 hours.

5. Soaking in gasoline for one month.

6. Soaking in gas engine oil for one month.

The result of these tests showed that panels soaked in gasoline or gas engine oil withstood the action of these substances better than if soaked in fresh water and that salt water had the same effect upon the glue as the fresh water. Specimens which withstood the boiling test invariably passed the baking test. Accordingly the tests retained for water-resistance qualities were the boiling and soaking tests in fresh water.

Boiling and Soaking Tests.--Panels to be tested were cut into specimens approximately five inches square. It was found that specimens which withstood 8 hours

boiling also withstood 24 hours boiling, hence for inspection purposes the eight hour boiling test was adopted. It was also determined that a specimen which withstood 8 hours boiling would apparently withstand two weeks soaking in cold water. Hence the boiling test was considered in the nature of an accelerated soaking tested, and, as an identical criterion, it was used in place of the soaking test because it could be very quickly completed.

The panels for test were made up of 1/16 inch veneer (generally birch faces and poplar core) glued, retained under pressure overnight and dried at 110°F. for 24 hours.

The designations adopted for the expression of results of the water-resistance tests on plywood specimens are given below:

Designation	Condition Corresponding
	ing
A	Indicates no separation of the plies.
B	Indicates that the total separation between all the plies, regardless of the number of plies, is not greater than 10 per cent of the area of sample tested.
C	Indicates a total separation greater than 10 per cent and not more than 50 per cent of the area of sample; or indicates blisters or other manufacturing defects of such nature as would warrant further tests.
D	Indicates a total separation of over 50 per cent of the area of sample tested.

Test for Adhesive Strength.--The test

for adhesive strength was made on a test

specimen of the form and dimensions shown

in Fig. 3. These specimens were made of

maple having a shearing strength of approx-

imately 2400 pounds per square inch. This

requires wood having a dry weight of about

50 pounds or more per cubic foot and a moisture content of 8 to 12 per cent. After gluing the test blocks, (from which the test specimens are subsequently cut) these were held in clamps under pressure for about 16 to 24 hours. They were then released from the clamps and allowed to stand for six additional days in order to dry and develop maximum strength. The test specimens were then cut to conform to Fig. 3 and tested in a standard Olson machine equipped with a special jig (shown in Fig. 4) to deliver a downward force against one member of the joint while the other rested upon a fixed platform.

This test is hereafter referred to as compression shear test or - simply shear test.

Test and Development of Formula VIII - #5

In the examination of formula VIII - #5, given below, it was decided to neglect the tests for adhesive qualities in this preliminary analysis until the proportions had been so adjusted as to give a glue of proper working qualities and consistently satisfactory ability to pass the boiling test.

```
100 gr. casein) Soak
300 gr. water ) 1/2 hour)
      +                  )
 25 gr. CaO   )          ))
125 gr. water )          ))
      +                  ) Mix
 50 gr. silicate         )
```

On preparation, the glue showed a very fluid consistency. Test panels of 1/16 inch stock (birch faces on poplar core - 3 ply) were made at intervals, as shown below, throughout the life of the glue (29 hours). These panels when tested gave consistently

satisfactory results, viz:-

Age of Glue When Tested	Boiling Test
0 Hrs.	A
1 "	A
1-1/2 "	A
3 "	A
4 "	A
6 "	A
7 "	A
8-1/2 "	A
22 "	A
24 "	A
26 "	A
27 "	A
29 "	A

On account of the too fluid charac-
ter of the above glue the silicate was re-
duced to 40 grams per 100 of casein. The
silicate was manipulated rather than the
water in order to reduce cost. This gave

a glue of slightly higher viscosity which
passed the boiling test satisfactorily but
showed a life of but 7-1/2 hours. In or-
der to further increase the viscosity, so
as to improve adhesiveness, it was decided
to manipulate the lime this time so as not
to jeopardize the glue-life any further.
The lime was then reduced to 20 parts of
CaO in place of 25. The result was a glue
of higher viscosity, greater apparent ad-
hesiveness, and of equally satisfactory
ability to pass test; its life was increased
to 13 hours by the reduction of lime.

The formula, by now, had apparently
matured to the point where it measured up
fairly satisfactorily to water-resistance
requirements when applied to the one case-
in used. It was next in order to apply
the formula to a series of other caseins.
The store of lime originally drawn upon

had now been exhausted. Its analysis
was unknown yet from its behavior and
appearance it was seen to be very much
air slacked and probably carbonated.
The new lime was much "quicker", hence,
higher in CaO. Since adherence to the
old proportions of 20 parts lime per
100 of casein·would lead to the intro-
duction of more actual CaO than inten-
ded, this difficulty was corrected ap-
proximately by reducing this figure to
25 parts per 200 of casein or 12-1/2
parts per 100 of casein. This approx-
imate procedure was considered justifi-
able inasmuch as the modifications so
far made were all very crude. The water
used for slaking the lime was also re-
duced to 90 parts per 100 of casein.

The modification of formula VIII -
#5 now presented the following appear-
ance:

Formula VIII - #10

```
100 gr. casein ) Soak
300 gr. water  ) 1/2 hour)
      +                  )
12-1/2 gr. quicklime )   ))
90 gr. water         )   ))
      +                  )Mix
40 gr. silicate          )
```

Formula VIII - #10 as given above, was
now applied to a series of caseins from the
behavior of which two features predominated.

1. The viscosities lacked any display
of uniformity, testifying again to the great
diversity in water-absorption abilities of
caseins.

2. The life of the various preparations
averaged but 4 to 5 hours.

The remedy proposed was to omit any re-
ference in the formula to the proportion of
soaking water - the latter to be determined
for each casein by trial, until further in-
formation became known. For case 2, the lime
and silicate were both manipulated in a direc-

tion which would increase the life.   This
led to the evolution of Formula No. 3.

Formula No. 3

```
100 gr. casein) Soak
 - 1 gr. water ) 1/2 hour)
     +                    )
  8 gr. CaO2 )            ) )
 90 gr. water )           ) )
     +                      ) Mix
 70 gr. silicate            )
```

Formula No. 3 was now applied to the
same series of caseins, which included
those prepared by the natural-sour, min-
eral acid and rennet processes.   The soak-
ing water was in each case determined by
trial as that quantity required to pro-
duce a viscous mixture of high adhesive
quality.   All of the glues passed the
boiling test satisfactorily.   Rennet case-
in required an abnormally large quantity

1 Proportions to be determined by trial

2 Equivalent to 10.6 gr. Ca (OH)$_2$

of water to produce the desired consistency; 280 parts of water were used for 100 parts of casein, or expressed as a ratio by weight of casein this equals 2.80.

Section IV - Development of
the Glue Formula

Proving of Formula No. 3

Glue No. 3 having presented promising aspects on application to a limited number of caseins, it was proposed to apply it to a wider range of caseins, whose histories of manufacture were known, in order to determine the possibilities of the formula. It was also hoped that reasons for the diverse character of caseins might become apparent from the data derived.

Caseins Used.--Through the cooperation of Mr. Dahlberg of the Bureau of Animal Industry, a series of caseins of a diversified

nature were prepared at the Grove City
(Pa.) Creamery and forwarded to the Labora-
tory for use in the following investi-
gation. In all, 25 caseins were stud-
ied, among them being a lactic casein
prepared by the writer, and a lactic case-
in obtained from the Knowles Creamery,
Knowles, Wis.

General Formula Employed.--Each case-
in was prepared in the form of a glue
according to formula No. 3 below, observ-
ing the necessary precautions regarding
water requirement. In a few instances
the glue viscosity for the same casein
was varied by changing the water content,
in order to determine what limit of water
content must be exceeded in order to make
manifest a significant depreciation of
properties.

```
100 parts casein) Soak
 - ¹parts water ) 1/2 hour)
        +                    )
10.6 parts powdered )        )
     hydrated lime )        ))
90 parts water      )        )) Mix
        +                    )
70 parts silicate            )
```

Preparation and Testing of Specimens.--
Within one hour after the preparation of
the glue, the test specimens were prepared.
These consisted of a veneer panel twelve
inches square, made up of 1/16" birch
faces and 1/16" poplar core, and two maple
shear blocks. These specimens were aged
and tested in the usual manner. The length
of life, viscosity, etc., of the glue were
recorded.

Results and Discussion.--The data de-
rived from the above procedure are recorded
in Table III. From its review there is in-
dication that caseins derived from ordinary
standard methods of manufacture, with the
possible exception of buttermilk caseins,
1-proportion water to be determined by trial

can be made to pass the boiling test
satisfactorily by a mere variation of the
amount of soaking water. From the con-
sistent failure in the soaking test, how-
ever, the formula is unsatisfactory. It
is to be noted in this regard that the in-
terchangeability of these two methods of
test for water-resistance quality can no
longer by considered as unqualified, i-
dentical criteria.

It is interesting to note that while
water absorption capacity[1] has heretofore
been observed to vary widely between the
limits of the range 1.3 to 2.8, the tabu-
lated data indicates very strikingly the
possibility of classifying caseins so as
to bring these limits closer together.
For lactic acid casein the ratio - soak-
ing water to casein by weight - varies

[1] Ratio of soaking water to casein by
weight.

in the range 1.35 to 1.7; for mineral-
acid casein in the range 1.7 to 2. plus;
for rennet casein previous work shows the
ratio has a value about 2.8.   In other
words the water absorption capacity is
dependent on some influence of the me-
thod of manufacture.

Examining into the influence of vis-
cosity upon the various glue properties
in order to observe the extent to which
necessity for high viscosity exists, it
is seen that low viscosities tend to
show inferior results in water-resistance
qualities as well as strength. A rela-
tively thick consistency, aside from in-
convenience of handling, seems more de-
sirable. The strength of the glue, when
viscous mixtures are used, appears to av-
erage 2400# / in.^{2}, comparing favorably
with high grade animal glue.

The glue life, for a fixed quantity of lime and silicate, depends on the viscosity; low viscosities yielding a longer life than high viscosities. Low viscosity mixtures are seen to attain a life of 90 hours whereas high viscosity mixtures show a life of only 20 hours.

It is to be noted that no one type of casein seems more desirable than others; all appear to be equally satisfactory.

Modification of Formula No. 3

In spite of the many desirable features of glues prepared according to Formula No. 3 the latter was absolutely unsatisfactory for the purpose in mind. Yet it was felt that the formula could be manipulated to pass the requirements of the soaking test by resorting to some simple modification.

Procedure.--The method of procedure,
for modification, was based upon the fact,
already known for sometime, that Glue No.
3, prepared without the addition of sili-
cate of soda, passed the cold water soak-
ing test satisfactorily. From this it
had been assumed that the silicate acts
counter to the calcium caseinate, either
by reacting with some of the lime and
hence depriving the casein of lime to this
extent, or else by the action of pepti-
zation. If the latter is the true explan-
ation, and if such action could not be
overcome, the use of silicate would have
to be rejected.

Working on the assumption, then, that
an insufficient quantity of lime is pre-
scribed in formula No. 3, it was proposed
to increase the lime content by two in-
crements; the first modification was to

use 13 parts lime per 100 of casein, the
second - 15 parts. These two modifica-
tions were to be applied to various case-
ins in order to ascertain the minimum
quantity of lime necessary to assure an
excess beyond that required to react with
both the casein and the silicate.

The eleven caseins used included
some commercial samples but were mostly
those furnished by Mr. Dahlberg; they
were fairly representative of the various
types ordinarily encountered.

With regard to the preparation of
the glues, it was anticipated that, since
higher lime content was now to be used,
high strength values, comparable with those
from the viscous mixtures previously used,
could be obtained by resorting to mixtures
of lower viscosity. Hence, the amount of
soaking water required in each case   was

judged as that necessary to impart to the
glue a consistency intermediate between a
thick, viscous mixture and a thin, watery
fluid; this consistency is hereafter des-
cribed as medium. Where the designations
+ or - are affixed to the term "medium"
it is intended to express a degree more
viscous or less viscous respectively than
is implied by the term "medium", etc.
Record was taken of such glue properties
as viscosity, life, etc.

The birch-poplar panel (1/16" stock)
specimens and the maple shear blocks were
prepared as usual, aged and tested. The
corresponding data appears in Table IV.

Results and Discussion.--Within the
range of caseins investigated, modifica-
tions I and II pass the boiling test equal-
ly well. Modification II, however, appears
more satisfactory in that practically all

of the glues prepared pass the cold wa-
ter soaking test perfectly. The strength
values compare satisfactorily with the high
values observed for the relatively more cum-
bersome, viscous mixtures of formula No. 3.
As was anticipated the increased lime caused
a material reduction in life, the latter
varying from 4 to 23 hours depending upon
the viscosity.

Casein VIIB exhibited abnormal behav-
ior due apparently to inferior quality.
It had a very offensive odor when prepared
as glue, and its long life was apparently
due to failure to gel because of hydroly-
tic action.

Modification No. II having different
properties from formula No. 3 is hereafter
designated as formula No. 4. It is presen-
ted below.

```
100 parts casein) Soak
 -  parts water ) 1/2 hour)
       +                  )
 15 parts powdered)        ))
    hydrated lime )        ))
 90 parts water   )        )) Mix
       +                   )
 70 parts silicate         )
```

Proving of Formula No. 4

The preceeding limited application of
formula No. 4, while it indicated that sat-
isfactory results could be obtained in gen-
eral, was not considered conclusive. Through
the efforts of Mr. Dahlberg, a very large
number of caseins had been prepared which
varied both in their methods of preparation
and in the factors affecting manufacture.
It was proposed to apply the formula to all
of the caseins available and to have the
caseins submitted to routine analysis. It
was hoped that from this great accumulation
of data relations would appear between the
glue-making qualities of caseins, their
methods of manufacture, their chemical analy-

ses, and their physical properties.

A new test designed to determine
shear strength of plywood had now been
perfected, as a supplement to the usual
method of tests, which it becomes neces-
sary to describe at this point.

Tension Shear Test.--It is readily
obvious that a shear test of plywood dif-
fers from the usual compression-shear tests
on maple specimens in that the grain of con-
tiguous layers lie at right angles to each
other, whereas in the case of maple shear
blocks parallel grain is encountered. Ac-
cordingly much lower strength values are
to be expected from plywood shear tests.

The specimens for this test are cut
(from the veneer panel) according to the
form and dimensions shown in Fig. 6.
These are then inserted in the jaws (Fig. 7)
of the Riehle testing machine shown in

Fig. 8. A schematic diagram illustrating
the operation of the latter is presented
in Fig. 9.

In using the machine the horizontal
beam carrying the load B and the counter-
weight W is levelled by regulating the a-
mount of shot in the hopper B. If shot is
removed from B the beam is thrown out of
balance by the action of the counterweight
W which causes depression on the right a-
bout the knife edge at A. The beam can now
be brought back into the horizontal by ap-
plying a downward force at C, (throughthe
aid of a handwheel) which is transmitted
back through the specimen and the system
of lever and links DEF to the beam, caus-
ing rotation about A in the opposite di-
rection.

In actual test the beam is brought
into the horizontal, the specimen inserted,

and a continuous stream of shot allowed
to discharge from B into the receptacle
A. The handwheel which delivers the
force at C is now put into rotation at a
rate sufficient to take up the yield in
the specimen and thus maintain the beam
horizontal. In this manner all of the
load due to the loss of shot from B is
thrown on the specimen. At rupture, the
corresponding load is read on the cali-
brated face of the scale at X which gives
the strength in shear in pounds per square
inch directly.

From the direction of the forces ap-
plied in this device to shear the specimen
the test is called tension-shear test.

Method of Procedure.--The procedure
followed in the application of formula
No. 4 to the various caseins was identical
with that described in the preliminary an-

alysis of the formula, veneer panel and shear-block specimens being prepared. Record was taken of the glue-life, viscosity, etc. The boiling and soaking specimens were cut into the form of tension-shear specimens in order that these would be in proper form for shear test after removal from water. Specimens for tension shear test in the dry state were also prepared.

The caseins used in the investigation numbered eighty six; seventy-three were furnished by Mr. Dahlberg; twelve were commercial samples, and one sample (rennet casein) was prepared by the writer. The lime employed was a high grade calcium lime analyzing practically 99% $Ca(OH)_2$. The silicate of soda used exhibited the following analysis:

Specific Gravity   1.38

Density (Beaume scale) 40.31°

Na_2O 9.38%

S_1O_2 30.50%

Results and Discussion.--The data derived from the investigation are arranged in Table V.

Study of this table shows that all of the caseins pass the boiling test perfectly. While the results of the soaking test are not quite so satisfactory, it appears that all caseins of reasonable purity will pass this test satisfactorily. It is consistently recorded that caseins of inferior quality, such as develop offensive odors when made into glue, will complete the test with a grade lower than B and hence are not satisfactory. These caseins, then, should be avoided. In general, with regard to water-resistance qualities, it may be said that

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formula No. 4 will produce satisfactory
glues with most caseins of a reasonable
degree of purity. Indication is that
special cases may be found in which some-
what more lime may be required. Such
special instances will have to be reme-
died according to the circumstances of
the case, whenever they occur.

The strength of the glue seems in-
dependent of the nature of the casein
used. Values in compression-shear aver-
age 2400# / in.^{2}, taking the nature of the
failure into consideration. The strength
in tension-shear averages about 300# / in.^{2}
with the type of jaws used. Since eccen-
tric loading is not entirely eliminated,
this value would undoubtedly be somewhat
higher with properly designed jaws. The
water saturated specimens are seen to
undergo a material reduction in strength,

boiled specimens being more seriously
affected than the soaked ones. It is
regretted that these water-saturated
specimens were not tested immediately
upon removal from water, in consequence
of which the values given are too high.
It is felt that a conservative estimate
of the average strength of the wet ply-
wood tested would be $150\# / $ in.^{2} for
soaked specimens and $100\# / $ in.^{2} for
boiled specimens.

Section V - Factors Responsible for Diverse
Character of Caseins

In the preceeding text it has been re-
peatedly pointed out that technical caseins
vary widely in their behavior; the annoy-
ances arising from this situation have like-
wise been indicated. With these facts in
mind, a comprehensive study of the data of
Table V reveals much information bearing

directly upon this immediate circumstance.
An analysis will readily confirm the fol-
lowing observations.

Influence of Manufacturing Conditions

On Chemical and Physical Nature.--
The chemical nature and physical proper-
ties of caseins from skim milk depend
chiefly upon the method of precipitation
and the subsequent treatment. Commercial
caseins containing the smallest amounts of
impurities (ash) are produced by the so-
called "grain-curd" method. This is a rel-
atively new process and has not yet been
widely adopted. It is capable of produc-
ing caseins as low in ash as one per cent,
although the average is probably nearer 1.8
per cent. Mineral acid caseins are relative-
ly higher in ash averaging probably in the
vicinity of 4 per cent. Natural-sour caseins
approach the low ash content of grain-curd

casein more closely with an average of
about 2.5 per cent, as ordinarily pre-
pared by the vat method. This relatively
low ash content, compared with mineral
acid caseins, is ascribable to the fact
that in the natural sour process the de-
velopment of acid is gradual and thus
occurs uniformly throughout the charge;
this local presence of acid combined with
the prolonged period of contact, typical
of the method, serves to dissolve from
the casein considerable of the mono-
calcium phosphate ($CaHPO_4$). The mineral
acid caseins on the contrary are in con-
tact with acid for a very limited period
of time and on account of the enmass pre-
cipitation of the curd, typical of this
method, efficient leaching of the impur-
ities is not possible. Rennet casein con-
tains, as a normal constituent the highest

ash content of all of these types of case-
in. The reason for this is due to the fact
that rennet casein contains combined cal-
cium, and, since it is prepared in a me-
dium free from acid any insoluble calcium
compounds mechanically held in the curd are
not removed.

Effect of Ash and Free Acid

The glue making qualities of caseins
are strongly influenced by the effects of
the conditions of manufacture as may be
seen from a consideration of the follow-
ing:

Using a fixed quantity of water, glues
prepared from caseins low in ash exhibit
lower viscosity and longer life than those
from caseins high in ash. This is striking-
ly evidenced (See Table VI) by the relatively
greater quantity of water required for min-
eral acid caseins in order to produce a com-

Table VI - Influence of Ash on Viscosity and Life

Type of Casein	Ash	Free[1] Acid	Ratio Soaking H₂O Casein	Consistency	Life
	%	ccs			hrs.
Rennet	8.39	-0.7	3.7	medium +	5
Sulphuric	5.10	4.4	2.25	stiff +	3
"	6.03	2.85	2.15	stiff-	4-1/2
"	5.36	1.8	1.85	stiff	6
"	4.29	1.35	1.8	medium	6-1/2
"	4.96	1.06	1.85	stiff	6-1/2
"	5.58	0.4	1.85	medium +	7
"	3.02	2.5	2.1	thin	18
"	2.64	-0.6	1.6	medium-	14
Lactic	2.62	2.8	1.65	medium +	6-1/2
"	2.51	4.0	1.55	medium	10
"	1.81	1.3	1.6	medium-	23

[1]Expressed in ccs of N/10 NaOH

parable viscosity and life, and is even
more pronounced in the case of rennet case-
in which normally contains the highest ash
content of any of the types of casein. It
is interesting, in this connection, to ob-
serve the following figures recently obtained
with a grain curd casein, the very low ash
content making necessary the use of the
smallest quantity of water so far used.

Type of casein	:% Ash	:Soaking: H_2O / casein	:Life (hrs.):	:Consistency
Grain curd	: 1.3	: 1.2	: 12	: medium

It is to be noted that, as the ash con-
tent of mineral-acid caseins approaches that
of a lactic acid casein, less water is re-
quired, and a lower viscosity and a longer
life are observed. In the case of lactic
caseins the higher the ash, the higher the
viscosity for a given water content and the
lower the life.

The influence of free acid does not appear to be sharply defined, inasmuch as caseins with a fixed quantity of ash manifest no consistant change either in viscosity or glue life with variation in free acidity.

Further evidence regarding the effect of ash is very strikingly manifested by the behavior of the same casein before and after washing. While both the ash and free acid contents are reduced by washing, apparently from what has already been noted, any change in glue behavior must be attributed to the change in ash content alone. Table VII shows that as the ash content is reduced by washing of the casein the glue viscosity for a fixed quantity of water is reduced and the life is increased. Furthermore because the viscosity is decreased less water is really required.

<u>Graphical Representation of Charac-</u>
teristics.--The general relations just
discussed are presented in a very strik-
ing manner, if a casein glue-character-
istic curve is constructed whose coor-
dinates are ash-content and water-absorp-
tion ability corresponding. In the graph
given (Fig. 10), four typical points,
selected from experience, are plotted -
one for each type discussed. The pro-
perties of each point have been so selec-
ted as to represent the average charac-
teristics observed for the type portrayed.
It will be noted that from the region of
low ash content to that of high ash con-
tent, the points lie practically along a
straight line curve, verifying the regu-
lar relation-ship which exists.

The data upon which the graph is based
are given in Table VIII. The ash content

Table VII.   Effect of Washing on Viscosity and Life

Type of Casein	No. of washings	Ash %	Free Acid ccs	(Ratio) Soaking H2O : casein	Consistency	Life hrs.
Lactic	0	2.51	4.0	1.55	medium	10
	3	1.44	2.6	1.50	medium -	18
"	0	2.46	3.6	1.45	medium +	7-1/2
	3	1.93	2.0	1.45	medium -	11
"	0	2.20	3.6	1.65	medium	11
	3	1.49	1.7	1.60	medium	16
"	0	2.62	2.8	1.65	medium +	6-1/2
	3	1.33	2.6	1.50	medium -	13
Sulphuric	1	5.58	0.4	1.85	medium +	7
	2	3.55	0.4	1.85	medium -	12
"	0	4.27	1.39	2.0	stiff	4-1/2
	2	5.36	1.80	1.85	stiff	6
"	0	4.40	0.1(3)	1.85	medium	6-1/2
	2	4.15	-0.1	1.85	medium -	9
"	0	3.94	1.4	1.7	medium	9
	several	2.64	-0.6	1.6	medium -	14

(3) The appearance of negative-acidity values is explained by VanSlyke as due to the fact that all of the casein is not base free-i.e., some calcium has come down with the curd in which it is present. Since no correction is made for this impurity all results on free-acidity are necessarily low and in some cases even negative.

has been selected to roughly represent
the average contamination peculiar to
specific types and the ratio, $\frac{water}{casein}$, is
such as to produce a medium viscosity
in the glue prepared from a casein of the
specific ash content given. In the ratio,
$\frac{water}{casein}$, by the term water is to be under-
stood the soaking water plus that added
with the lime, i.e., total water.

Table VIII - Data for Casein Glue-
Characteristic Curve.

Type of Casein	: Ash %	: (Ratio) water casein
Grain-curd	: 1.8	: 2.3
Natural sour:	2.5	: 2.45
(Ordinary) Mineral acid:	4.0	: 2.8
Rennet	: 8.6	: 3.9

The value of such a graph for the pur-
pose of pre-determining the quantity of

water which will be required for a
specific casein of known ash content,
when made into a glue according to for-
mula No. 4, is verified by checking the
curve against the ash content and water
absorption ability of the caseins described
in any of the accompanying tables. In
such a comparison it is well to bear in
mind that the water content recorded must
be corrected to conform to a medium vis-
cosity, wherever such a viscosity did not
actually exist, inasmuch as the graph was
constructed for glue preparations of medium
viscosities.

For example, from Table VI

Per cent Ash   6.03

$\dfrac{\text{Water}}{\text{Casein}}$ 3.05

Consistency   Stiff -

These observations were not made on a glue
of medium viscosity, hence the water re-

quirement must be changed to be comparable
with the curve. The corrections to be ad-
ded in any case, to the ratio, $\frac{water}{casein}$, when-
ever the viscosity recorded is not medium,
are given below for the corresponding **vis-
cosities** shown.

thin	- 0.4 parts
medium -	- 0.2 "
medium +	+ 0.2 "
stiff	+ 0.3 "

Making the proper correction, the ratio,
$\frac{water}{casein}$, in the above extract from Table VI
becomes 3.25. On reference to the graph, it
is seen that this figure checks the graph.

It is to be expected that slight dis-
crepancies between observed requirements
and those prescribed by the graph will be
encountered since the observation of con-
sistency is but approximate at best.

If the curve be continued by extending
the straight line in the dotted portion shown,

caseins which do not fall within the range
actually investigated may be included in
its scope by this extrapolation. It may
now be predicted that a pure casein, free
from ash would require about 1.85 parts of
water. The nearest approach to such a
casein is one recently made available from
the laboratory of VanSlyke. Its ash content
determined in platinum was 0.38 per cent.
The graph prescribes that such a casein
requires 1.95 parts water per part of case-
in to form a glue of medium viscosity. On
actual test 2.1 parts were required.

It is now possible by deriving the e-
quation of the straight line graph to make
available a mathematical relation between
the ash content of a casein and its cor-
responding $\frac{water}{casein}$, ratio. For simplicity
in the following discussion the ratio,
$\frac{water}{casein}$, is designated by W. Writing the

equation of a straight line as $y = mx + c$,
the constants may be evaluated from the fol-
lowing considerations. From the graph, a
casein of 0.0 per cent ash content has a
value of W equal to 1.85, and the slope of
the line being observed by simple inspec-
tion to be 0.24 the equation becomes:

$Y = W = 0.24x + 1.85$, where x equals
per cent ash content of the casein.

BIBLIOGRAPHY ON CASEIN AND CASEIN GLUES

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the literature, as yet, on the special sub-
ject of water-resistant glues. By selecting
from the following bibliography much infor-
mation regarding casein, its properties,
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obtained;occasionally references to the use
of casein in glue preparations also occur.

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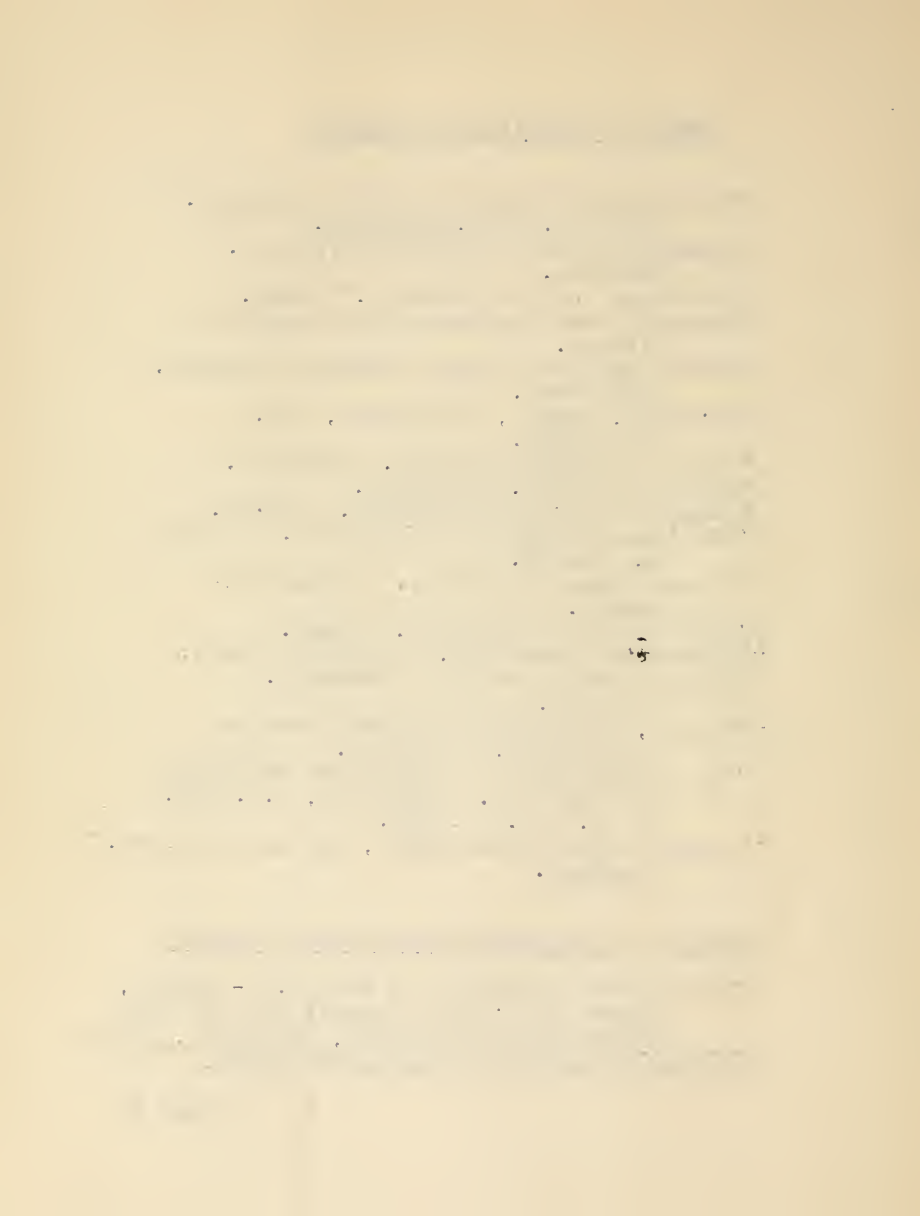
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             845,790)   Isaacs, 1906
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             609,200   Hall, 1896 (Reissue
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A large number of articles have been
published on the chemical properties of
pure casein. They can be located through

"Chemical Abstracts." Most of the articles published in English have appeared in the following journals:

> Journal of Biological Chemistry
> Journal of Physical Chemistry
> Journal of the American Chemical
>   Society
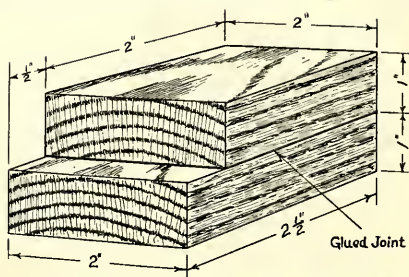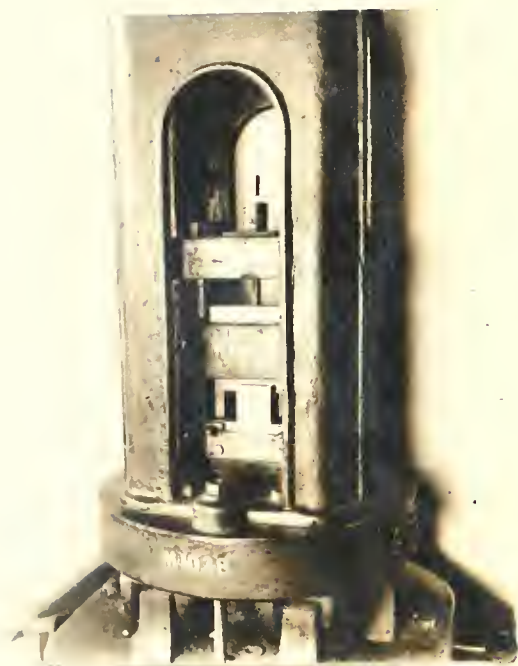> Technical Bulletins of the New York
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Fig. 3 —Standard Test Specimen for Testing Adhesiveness.
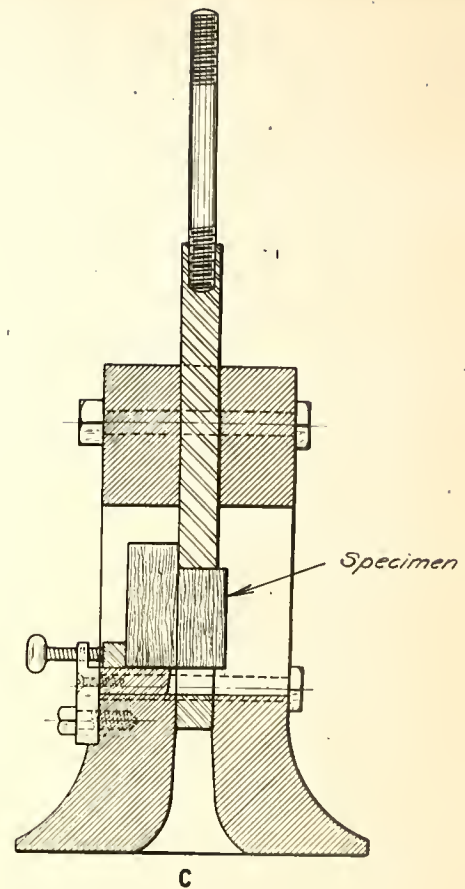
A

6239 M.

B

Glued Joint
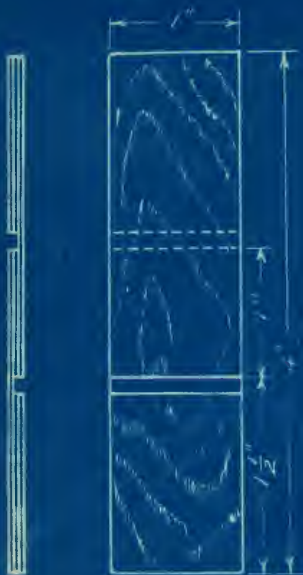
C
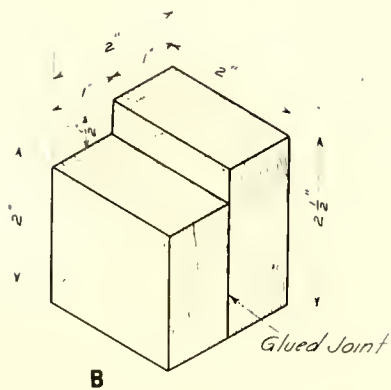
Specimen

Fig. 4

FIG. 6

FIG. 7

PLYWOOD and SPECIMEN
METHOD OF TESTING

FIG. 8

Fig. 9.

Schematic Drawing of Riehle Machine

Ash Water Absorption Curve
for
Casein Water Resistant Glues
(for medium-viscosity glues)
GLUE FORMULA 4A
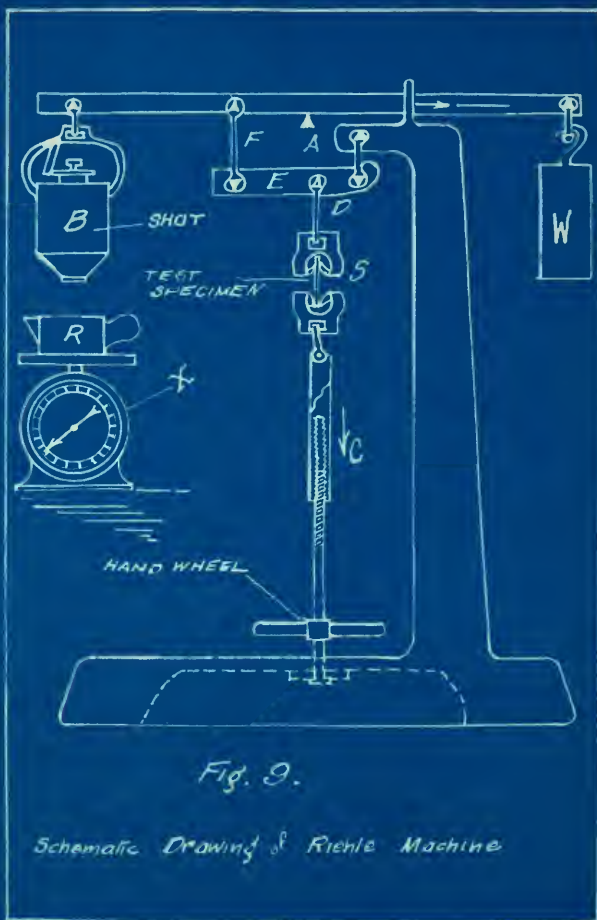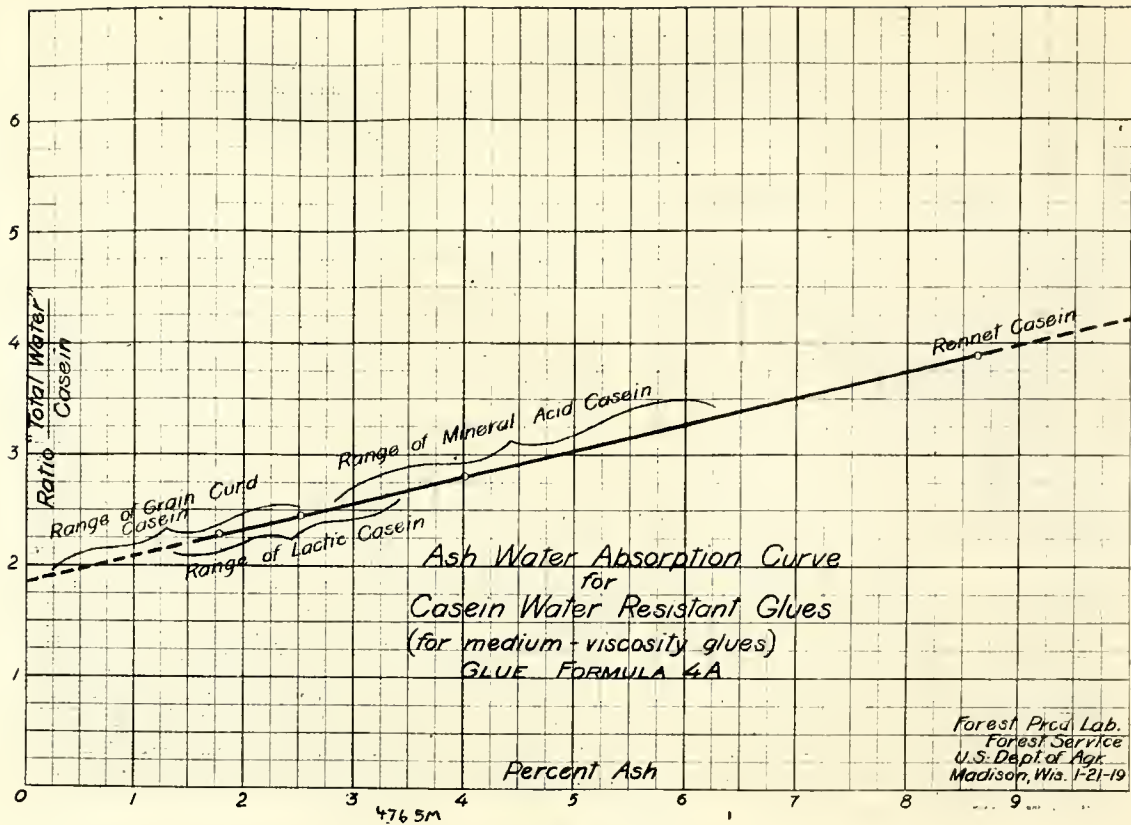
Fig. 10

Table III - Results of tests with formula No. 3.

Sample	Manufacture	Ration (by weight) Soaking water to Casein	Corresponding Consistency	Life in Hours	Boiling Test	Soaking Test	Strength in Shear Maximum Lbs.per sq. in.	Minimum Lbs.per sq. in.	Mean Lbs.per sq. in.	Nature of Failure % Wood Maximum	Minimum
96C (a)	Ejector Method	1.55	Stiff	20	A	D	3580	2790	3000	85	75
95A	" " "	1.55	"	30+	A	D	2860	1930	2599	70	10
95B	" " "	1.55	"	35+	A	D	1840	2380	3701	75	80
33E	" " "	1.60	Medium +	55+	A	D	----	----	----	--	--
XV-E	(b) Lactic (c)	1.35	Stiff	50+	A	D	2625	2095	2396	0.0	10
XVI-E	(b) Lactic (Knowles)	1.35	" -	50+	A	D	2900	2250	2634	0.0	0.0
59-C	Ejector Method	2.60	Watery	58+	A	D	2120	1695	1955	0.0	0.0
59-C 1	" "	2.00	Medium -	63+	A	D	3020	2450	2667	10	10
59-C 2	" "	1.70	" -	55+	A	D	3110	2345	2777	90	50
59-C 3	" "	2.00	" -	60+	B	D	2345	1977	2185	0.0	0.0
61-D	" "	1.70	" -	61+	A	D	2940	2195	2624	100	10
61-D 2	" "	1.75	Thick	32+	A	D	3295	2650	2950	10	100
34-B	" "	1.75	"	--	A	D	----	----	----	--	---
B 2	" "	1.75	Medium -	90	D	D	----	----	----	--	---
60-C	" " "	1.60	" +	40+	A	D	2800	2600	2716	90	50
C 2	" "	1.60	" +	43+	A	D	----	----	----	--	--
C 4	" "	1.60	Stiff -	60	A	D	----	----	----	--	--
22	Ejector Butter- milk	1.75	Medium -	88+	D	D	2925	2925	2701	0.0	0.0
61-C	HCl curd cook- od 158° F.	2.00	Stiff -	20 (appr)	A	D	2810	1975	2249	0.0	0.0
80-C 2	HCl curd cook- ed 146° F.	1.95	Thick +	--	A	D	2920	2650	2770	50	30
34-A	H₂SO₄	1.75	Thick	40+	A	D	2865	2245	2472	0.0	0.0
33-A	"	1.75	" -	68+	A	D	----	----	----	--	--
80-A	"	1.75	" +	18	A	D	2820	2735	2780	100	30
96-A	"	1.85	"	28+	A	D	2155	1775	1958	70	40
60-A	"	1.75	Very thick	20	A	D	2270	2150	2219	100	60
B 2	"	1.75	" "	20	A	D	3080	2985	3016	0.0	50
B 3	"	1.75	" "	--	A	D	3055	2320	2677	80	100
61-A	"	2.00	Medium	50+	B	D	2890	2525	2691	50	75
61-A 2	"	1.70	Thick	--	A	D	2930	2535	2675	60	60
59-A	"	2.60	Very watery	81+	B	D	958	907	918	0.0	0.0
A 2	"	1.75	Thick	20	A	D	3200	3075	3166	10	100
A 2	"	1.75	"	20	A	D	2840	2580	2760	90	100
80-B	H₂SO₄ curd cook- ed 166° F.	1.75	Thick +	20	A	D	3330	2870	3180	60	80
60-A	H₂SO₄ curd cook- ed 175° F.	1.75	" +	20	A	D	2460	1845	2170	70	10
96-B	H₂SO₄ curd cook-ed 175° F.	1.85	"	32+	A	D	3120	2420	2780	60	10
59-B	H₂SO₄ curd cook- ed 170° F.	2.60	Watery	80+	A	D	2380	2145	2215	0.0	.40
B 2	" " "	2.00	Medium	55	A	D	2560	2200	2332	90	.10
B 3	" " "	2.00	"	50+	A	D	3530	3295	3366	70	50

Note:

The designations + and - which appear in the column headed "Corresponding Consistency" signify a degree slightly greater or less respectively than preceding term.

The designation + which appears in the column headed "Life in Hours" indicates that gelatinization occurred a few hours later than indicated by the figure recorded.

(a) Lactic souring.
(b) Had very sour cheesy odor due to improper drying which had caused slight putrefaction.
(c) Prepared by writer.

7365 M.

Table IV - Results on tests of two modifications of glue No. 3.

	Casein							Strength in Shear					
Mark	Method of Manufacture	Consistency	Soaking Water: Casein	Life	Boiling Test	Soaking Test	No.of Specimens Tested	Wood Maximum Lbs.per sq. in.	Wood Failure %	Minimum Lbs.per sq. in.	Wood Failure %	Average Lbs. per sq.in	Wood Failure %
						MODIFICATION I.							
VIII-D:	----	Thin	1.7	27 hrs.	A	A	8	2300	90	1660	60	2008	90
97 B:	Lactic	Medium -	1.45	16 +	A	A	8	3040	75	2550	70	2816	40
59 A:	H$_2$SO$_4$	Thick -	1.75	5	A	A	4	2660	60	2550	40	2600	49
33 A:	H$_2$SO$_4$	Medium +	1.7	22	A	A	8	2630	10	2320	10	2482	40
96 C:	Lactic	Medium	1.55	20 +	"	"	4	2600	30	1940	20	2356	20
VII-B:	----	Medium	1.9	144 +	"	D	4	2500	20	2190	20	2242	20
60 C:	Lactic	Medium	1.6	29	"	B	8	2280	100	1940	100	2125	91
97 D:	Lactic	Medium	1.6	29	"	A	4	2690	0	2050	60	2505	20
95 B:	Lactic	Medium	1.55	20	"	A	4	2560	85	2175	10	2400	74
95 A:	Lactic	Medium +	1.55	12	"	A	4	2565	20	2450	10	2481	15
97 C:	Lactic	Medium	1.65	18	"	A	4	2500	70	2275	10	2390	41
						MODIFICATION II							
VIII-D:	----	Thin +	1.65	12	A	A	4	2690	90	2320	50	2440	57
97 B:	Lactic	Medium -	1.45	11	"	"	4	2220	0	2795	0	2862	0
59 A:	H$_2$SO$_4$	Thick +	1.65	4	"	"	8	2775	10	2300	0	2481	0
33 A:	H$_2$SO$_4$	Medium +	1.7	10	"	"	8	2680	40	2200	0	2486	39
96 C:	Lactic	Medium -	1.55	10	"	"	4	2560	10	2495	40	2540	15
VII-B:	----	Medium	1.9	192	"	B	8	2680	0	2180	10	2501	42
60 C:	Lactic	Medium -	1.6	23	"	A	8	2725	10	1900	25	2435	60
97 D:	Lactic	Medium -	1.6	16	"	A	8	2510	0	2000	10	2285	40
95 B:	Lactic	Medium -	1.5	18	"	A	4	2560	60	2100	85	2400	60
95 A:	Lactic	Medium	1.55	10	"	A	8	2940	10	2040	80	2200	98
97 C:	Lactic	Medium -	1.65	11	"	A	8	2710	30	2075	80	2455	60

7364 M

TABLE No. V - APPLICATION OF FORMULA-4-TO-DAHLBERG CASEINS ‡

CASEIN

No.	Type	Object of Manufacture	Conditions of Manufacture: Acidity before Precipitation #	Fat	Acid added Conc. ccs/100 # Milk	Temperature of Precipitation	Temperature of Curd Cooked	Number of Washings	Drying Temperature	Drying Period	Analysis: Color	Odor	Moisture Per cent	Fat Per cent (Moisture Free Basis)	Ash Per cent	Free Acid in ccs N/10 NaOH	Nitrogen M.F.A. Free
XX 5	—	—									White	Offensive	7.25%	%.77	4.9%	10	14.97%
XX D	—	—						+			Yellow		6.30	2.17	3.93	33	13.64
33 A	H_2SO_4		.21%		474	49°C		1	545°C	5 Hrs	Yellow	Sour	8.30	6.32	3.34	2.20	14.60
XX C	Rennet	For Experimental Purposes				545°		6	545	8	White	None	334	.83	8.39	0.8	14.32
39 A	H_2SO_4	Comparison Acid vs	.18	02	50	320		0	545	6	Slightly Yellow	—	907	.30	4.87	2.3	14.09
39 C	Ejector	Ejector Method	.96	02		335		3	549	6	White	—	944	.39	1.93	.18	15.11
	-											—					
60 C	Ejector		.80	.03		335		3	535	5	White	Sour	8.92	.34	1.81	1.30	1304
61 D	Ejector		95	.02		35		3	545	6	White	Sour	8.89	.34	1.81	1.80	1490
95 A	Ejector	Effect of Washing on Curd Precipitation	.91	tr		60		0	51	8	White	Sour	8.77	.01	2.31	4.0	1438
95 B	Ejector	With Excess Acid	91	tr		60		3	51	8	White	—	892	.03	1.44	26	13.17
96 B	H_2SO_4 Cooked	Comparison Cooked Curd and	.18	01	30	49	80	1	51	8	White	—	942	.02	4.29	1.35	1491
96 C	Ejector	Ejector Methods	76	01		60		3	51	8	White	—	910	.05	2.09	2.0	1581
97 A	Ejector	Effect of Washing on	72	01		54½		0	52	9	White	Sour	6.70	.17	2.46	36	1475
	-	Lactic Curds Precipitated at															
97 B	-	Low and High Acid	72	01		545		3	52	9	White	Slightly Sour	7.39	.07	1.93	2.0	13.39
97 C	-	Concentrations	98	01		545		0	52	9	White	—	6.87	.91	2.20	36	1434
97 D	-		98	01		545		3	52	9	White	—	708	.16	1.49	17	1492
75	—	— —									White	Very Sour	7.89	3.78	10.01	0.3	1338
93 A	Ejector	Ejector Method	82			462		2	575	8	White	—	7.50	.14	1.62	30	1492
-	-	vs															
-	-																
98 B	Lactic Vat	Cottage Cheese Method	.81			325		2	575	8	Yellow	—	6.77	.13	2.18	44	1469
-	Method																
99 A	H_2SO_4	Study of Influence of	.20		60	520		1	575	—	Yellow	—	666	.05	4.91	20	1450
99 D	-	High Precipitating Temperature	.20		625	66		1	575	—	White	—	665	.04	4.96	106	1442
100 A	H_2SO_4		.18	003	85	525		1	—	6	White	—	854	.12	4.39	2.0	1455
100 B	-	Influence of Excess	.18	003	70	525		1	—	6	White	—	844	.05	3.86	21	1460
-	-	Acid for Precipitation															
101 A	H_2SO_4 Cooked		.24	01	92	336	77	2	52	6	White	—	876	.14	3.55	.4	1540
-	-	Influence of Quantity															
101 B	-	of Acid Used for	.24	01	40	326	80	1	52	6	White	—	872	.04	3.38	4	1498
-	-	Precipitation															
102 A	H_2SO_4	Effect of Washing	.23	01	316	336		0	49	5	White	Sour	896	.112	4.27	139	1445
102 B	-		.23	01	316	336		2	49	5	White	Sour	860	0.10	3.36	1.80	1432
103 A	H_2SO_4	H_2SO_4 Method	.20		420	325		1	52	6	White	Slightly Sour	819	0.05	4.82	2.92	1436
		vs															
103 B	HCl	HCl Method	.20		96	525		1	52	6	White	Sour	836	0.08	6.09	2.83	1420
104 A	H_2SO_4	Effect of High	.16	.003	30	545		0	52	5	White	—	278	0.08	5.08	4.18	1422
-	-	Drying Temperature															
104 B	-	Upon Washed and	16	.003	30	545		0	636	—	Very Yellow	—	439	0.16	5.10	4.40	1420
104 C	-	Unwashed H_2SO_4 Curd	16	.003	30	545		2	520	5	White	Sour	683	0.07	4.82	4.30	1428
104 D	-		16	.003	50	545		2	656	4	Slightly Yellow	Sour	4.15	0.10	4.99	3.92	1435
105	Lactic H_2SO_4	Effect on Ash Semi-Sour Milk	30	.01	15	38		2	52	7.5	Slightly Yellow	None	7.50	0.34	2.69	3.50	1437
			20	.003	50	545		2	52	4	White	—	737	0.15	3.06	1.9	1430
106 A	H_2SO_4	Effect of Time	.20	.005	30	549		2	52	4	White	None	257	0.15	3.06	1.9	1430
106 B	-	of Drying	20	.003	50	545		2	528	8	White	None	722	0.13	3.07	1.4	1438
107 A	H_2SO_4	Effect of High	20	01	50	545		2	52	4	White	None	794	0.16	4.72	-0.7	1487
		Drying Temperature on															
107 B	-	Washed and Unwashed	.20	01	50	545		2	66	6	White	None	728	0.15	4.71	-0.6	1460
107 C		H_2SO_4 Curds	.20	01	50	545		0	52	4	White	None	7.70	0.17	4.70	0.1	1417
107 D	-		.20	01	50	545		0	66	6	White	None	711	0.19	4.78	1.8	1383
108 A	H_2SO_4	Effect of Excess Acid in	19	01	100	545		0	32	69	White	None	777	0.50	2.99	30	1453
		Precipitation															
108 B		Effect of Excess Acid and High Temperature [illegible]	.19	01	100	66		0	32	65	White	None	779	0.37	3.02	2.50	1451
109 A	Lactic	Effect of Washing	72	01	-	32		3	52	10	White	None	750	0.06	1.33	2.60	1475
109 B	-	Natural Sour Curd	75	01		52		0	52	10	White	None	736	0.31	2.82	2.8	1470
110 A	H_2SO_4	Effect of High Precipitating Temperature	19	.005	50	545		1	54	7	White	None	833	0.14	3.06	0.2	1479
110 B	-	and Excess H_2SO_4	.19	.003	73	60		1	54	7	White	None	813	0.10	3.78	1.2	1469
111 A	H_2SO_4 Cooked	Effect of Excess	20	01	90	545	77	0	545	6	White	None	768	0.08	4.40	0.1	1430
111 B	-	H_2SO_4	20	01	666	545	77	2	545	6	White	None	246	0.12	4.19	-0.1	1498
112	H_2SO_4	Routine Creamery Run	.19	.01	46	545		2	520	7	White	None	765	0.06	4.96	2.7	1430
113 A	HCl	Curd Allowed to	.19		129	545		2	52	8	White	Slightly Sour	762	0.23	3.65	0.46	1438
113 B	-	Decompose before	.19		129	549		0	32	8	White	Slightly Sour	6.84	0.14	3.84	-0.4	1402
113 C	-	Drying	.19		129	549		2	Shipped and allowed to rot		Gray	Offensive	675	0.16	3.51	-0.8	1477
113 D	-		.19		129	545		0	Several days		White	Sour	694	0.14	3.73	1.6	1451
114	Lactic H_2SO_4		.36	.003	24½	345		2	52	8	Slightly Yellow	None	294	0.17	3.29	0.7	1492
115 A	H_2SO_4	Effect of Precipitating	.19	01	36¼	31		1	52	7	White	None	696	0.10	4.37	-0.2	1350
115 B	-	Temperature	.19	01	36¼	38		1	52	7	White	None	706	0.07	4.68	-0.4	1438
116	H_2SO_4 HCl		.19	01	3¼ + 68¾	545		2	52	7	White	None	6.88	0.25	4.75	1.2	1440
117	H_2SO_4		17	.015	666	32		2	52	7	White	None	7.13	0.44	3.98	0.0	1460
118	Ejector		65	01		33		2	52	7	White	None	7.69	0.26	2.52	0.0	13.10
119	HCl		.19	01	136 (3¼ HCl)	335		0	545	7	Slightly Yellow	None	8.32	0.21	3.70	1.3	1408
121 A	Ejector	Effect of	.80	01		345		0	52	10	White	None	8.08	0.10	2.90	1.3	1459
121 B	-	Washing Ground Curd	80	01		545		(b) 4+4	52	10	White	None	847	0.25	1.73	0.1	1340
122 A	Lactic H_2SO_4 Cooked	Influence of Development of	20	02	60	545	85	1	49	5	Gray	None	859	0.10	3.38	-1.4	15.19
122 E	-	Lactic Acid	33	02	20	325	85(2)	1	49	5	White	Slightly Sour	813	0.11	4.53	-1.4	15.20
123 A	-	Influence of Development	20	05	60	32	85(2)	1	49	7			791	0.26	3.65	-0.9	13.21
123 C	-	of	.33	05	32	32	85	1	49	7	White	None	926	0.11	4.60	-1.7	13.50
123 E	-	Lactic Acid	42	05	10	49	85	1	49	7	White	None	909	0.13	4.56	-1.6	13.40
124 A	H_2SO_4	Effect of Washing	20	02	63	50		0	49	10	Slightly Yellow	None	7.43	0.07	3.94	1.4	1414
124 B	-	Ground Curd	20	02	63	50		Several	49	10	White	None	690	0.10	2.64	-0.6	15.08
125 A	Lactic H_2SO_4 Cooked	Influence of Development	.18	.025	54	545	85(4)	1	49	7	White	None	935	0.14	3.78	-2.6	13.38
125 B	-	of Lactic Acid	43	.025	6	48	85(4)	1	49	7	White	None	910	0.10	4.66	-2.2	13.52
126 A	Lactic HCl Cooked		.18	.019	130	51	85	1	49	8	White	Sour	962	0.22	4.60	-0.9	15.08
126 B	-	Influence of Development	26	.019	100	51	85	1	49	8	White	Slightly Sour	921	0.22	4.56	-1.3	13.31
126 C	-	of Lactic Acid	32	.019	80	52	85	1	49	8	White	None	932	0.19	4.89	-1.9	14.61
126 D	-		30	.013	30	50	85	1	49	8	White	Slightly Sour	882	0.19	4.14	-1.3	13.27
127	HCl Cooked Curd	Influence of Low Precipitating Temperature	.20	.015	[illegible] reached	348	85	1	49	7	White	Sour	917	0.13	4.48	-0.6	13.17
128 A	HCl Cooked Curd		20	.01	140	49	85	1	49	6	White	None	917	0.10	4.80	-1.4	14.88
31	Dunhams	HCl									White	Sour	776	1.04	4.40	4.15	14.28
32	-	H_2SO_4 .13									White	Sour	726	1.83	4.06	2.83	14.64
33	-	Lactic Acid									White	Slightly Sour	763	0.47	3.67	3.13	14.60
34	-	H_2SO_4 - 93									White	Offensive	722	1.56	4.79	3.33	14.74

GLUE

No.	Ratio Soaking Water / Casein	Consistency	Life in Hrs	Remarks	Boiling Test (8 Hrs) *	Soaking Test (10 Days) *
XX 5	19	Medium	192	Offensive Odor	A	D
XX D	165	Thin+	12		A	A
33 A	1.7	Medium+	10		A	A
XX C	2.8	Medium•	5		A	A
39 A	165	Thick+	4		A	A
39 C	1.30	Medium-	11		A	C
	130	Medium-	—		A	A
60 C	16	Medium-	23		A	A
61 D	15	Medium	12+		A	A
95 A	135	Medium	10		A	A
95 B	15	Medium-	18		A	A
96 B	18	Medium	6½		A	A
96 C	135	Medium-	10		A	A
97 A	145	Medium•	7½		A	C
	145	Medium•	6½		A	A
97 B	145	Medium-	11		A	A
97 C	165	Medium-	11		A	A
97 D	16	Medium-	16		A	A
75	14	Medium	6		A	A
93 A	13	Medium•	12		A	A
-	13	Medium	8½		A	D
-	13	Medium	8		A	A
98 B	13	Medium	7	Lumpy-Yellow	A	D
-	13	Medium	—		A	A
99 A	185	Stiff-	5½		A	A
99 D	185	Stiff	6½		A	A
100 A	185	Medium	7½		A	A
100 B	185	Medium-	8½+		A	D
-	185.	Medium-	7½		A	A
101 A	185	Medium-	12	Lumpy	A	D
-	185	Medium-	—		A	A
101 B	185	Medium+	7		A	D
-	185	Medium+	—		A	A
102 A	20	Stiff	4½		A	A
102 B	185	Stiff	6		A	A
103 A	185	Stiff-	7½		A	D
	183	Medium+	—		A	D
103 B	2.15	Stiff-	4½		A	A
104 A	2.0	Medium•	6½		A	D
-	20	Medium•	—		A	A
104 B	225	Stiff	3	[illegible]	A	A
104 C	2.0	Medium	9		A	A
104 D	2.0	Medium+	9		A	A
105	1.75	Medium-	18		A	A
						A
106 A	185	Stiff-	7		A	A
106 B	2.10	Medium	8		A	A
						A
107 A	2.0	Medium	9½		A	A
						A
107 B	20	Medium•	8		A	A
107 C	21	Medium	7½		A	A
107 D	21	Medium+	6½		A	A
						A
108 A	21	Thin	20		A	A
						A
108 B	21	Thin	18		A	A
109 A	1.5	Medium-	12		A	A
109 B	165	Medium+	6½	Very Lumpy Brown	A	A
110 A	20	Medium	6		A	A
110 B	20	Watery	10		A	A
111 A	185	Medium	6½		A	A
111 B	18	Medium-	9		A	A
112	18	Medium+	5		A	A
113 A	2.15	Medium	8		A	A
113 B	2.15	Stiff-	4½		A	A
113 C	1.35	Medium-	30+	[illegible]	A	D
113 D	1.70	Medium	10	Casein decomposed	A	D
114	20	Stiff-	4		A	A
115 A	18	Medium+	4½		A	A
115 B	18	Medium+	4½		A	A
116	2.0	Medium	7		A	A
117	15	Medium+	8		A	A
118	17	Medium	9		A	A
119	195	Stiff	5		A	B
121 A	1.3	Medium	13		A	A
121 B	14	Medium	16		A	A
122 A	195	Medium	10		A	A
122 E	165	Medium	6		A	A
123 A	1.6	Medium	12		A	A
123 C	155	Medium+	8		A	A
123 E	175	Medium+	7		A	D
124 A	1.7	Medium	9		A	A
124 B	1.6	Medium-	14		A	D
125 A	18	Medium	7½		A	A
125 B	1.9	Medium+	6		A	A
126 A	185	Stiff-	6		A	A
126 B	1.9	Medium	12		A	A
126 C	18	Medium	8		A	A
126 D	1.7	Medium	10		A	D
127	2.0	Medium	7		A	A
128 A	1.6	Medium•	5		A	A
31	1.9	Medium	9+		A	D
32	1.75	Medium	9+		A	D
33	1.7	Medium+	9+		A	B
34	1.85	Medium+	288+	Offensive Odor	A	D

TESTS

No.	Tension Shear Lbs/in²: Dry Panel Max	Failure	Min	Failure	Ave	Failure	Panel Boiled (8 Hrs) Max	Failure	Min	Failure	Ave	Failure	Panel Soaked (10 Days) Max	Failure	Min	Failure	Ave	Failure	Compression Shear Lbs/in²: Lbs Max	Per Cent Wood Failure	Lbs Min	Per Cent Wood Failure	Lbs Ave	Per Cent Wood Failure
XX 5																			2680	0%	2180	10%	2301	42%
XX D																			2690	90	2320	50	2440	40
33 A																			2680	40	2200	0	2486	39
XX C	270	Glue	245	Glue	237	Glue	175	Glue	100	Glue	149	Glue	190	Glue	160	Glue	173	Glue	2262	100	1600	30	1917	60
39 A																			2775	10	2300	0	2481	10
39 C																			2962	100	2525	70	2442	20
	235	Face	186	Face	233	Face	180	G+C	130	Glue	157	Glue	165	Glue	150	Glue	154	Glue	2450	85	2250	75	2381	90
60 C																			2725	100	1900	25	2435	60
61 D					304	Face	183	Glue	180	Glue	182	Glue							2260	100	1660	100	1920	83
95 A																			2340	100	2040	80	2200	98
95 B																			2960	60	2100	85	2400	60
96 B	305	Core	393	Core	355	Core							195	Glue	185	Glue	195	Glue	2325	84	2000	72	2339	64
96 C																			2560	10	2499	40	2540	15
97 A																								
	280	Face	255	Face	265	Face	205	Glue	185	Glue	197	G+C	220	G+C	200	G+C	210	G+C	2575	95	1800	90	2212	80
97 B																			2920	0	2799	0	2862	0
97 C																			2710	30	2075	80	2445	60
97 D																			2910	0	2000	10	2289	40
75	310	Face	275	Face	271	Face	180	Glue	129	Glue	166	Glue	280	Face	160	Glue	228	G+F	2090	100	1850	100	1848	90
93 A	245	Face	199	Face	239	Face	180	G+C	100	Glue	191	G+C	203	Core	120	Core	172	Core	3050	50	2490	70	2602	96
-	360	Core	315	Face	338	C+F													2475	100	2373	10	2431	60
-	410	Glue	295	Face	393	G+F	130	Glue	130	Glue	143	Glue	200	Glue	175	Glue	186	Glue	2379	50	1987	80	2286	78
98 B	295	Face	335	Glue	233	C+F													2787	50	2029	90	2380	43
-																			2762	100	1712	100	2307	44
99 A	300	Core	290	Core	296	Core							298	Glue	250	Glue	271	Glue	2905	95	2800	15	2649	91
99 D	290	Core	194	Face	233	F+C							320	Core	285	Glue	296	G+C	2899	98	2575	100	2709	80
100 A	360	Core	319	Face	334	F+C							290	Glue	220	Glue	267	Glue	2550	100	2340	100	2474	100
100 B	295	Face	235	Glue	233	Face							170	Glue	125	Glue	138	Glue	2370	90	1875	82	2178	64
-	260	Face	210	Face	233	Face	190	Glue	180	G+C	183	G+C	183	Glue	180	Glue	190	Glue	2700	60	2487	100	2606	74
101 A	315	Core	220	G+C	272	Core							200	Glue	100	Glue	146	Glue	2425	100	1973	40	2129	82
-	300	Face	240	Face	292	Face	173	Glue	80	Glue	146	Glue	235	G+C	210	G+C	211	G+C	2675	23	2390	100	2447	60
101 B	330	Core	300	G+C	310	Core	195	Glue	125	Glue	166	Glue							2425	5	2300	10	2335	7
-	335	Face	235	Face	292	Face	180	G+C	160	Glue	175	G+C	230	G+C	210	G+C	222	G+C	2330	100	2112	100	2249	100
102 A	285	Core	250	Glue	267	G+C							170	Glue	115	Glue	146	Glue	2940	0	2670	100	2801	39
102 B	250	Core	225	Core	232	Core							280	Glue	200	Glue	231	Glue	2649	100	2343	95	2448	78
103 A	290	Core	247	Core	266	Core							140	Glue	75	Glue	115	Glue	2720	85	2110	99	2381	78
	344	Face	305	Face	318	Face	220	G+C	185	Glue	201	Glue	150	Glue	140	Glue	145	Glue	2437	100	2087	100	2236	100
103 B	295	Core	265	Core	281	Core	299	Glue	248	Glue	250	Glue							2545	5	2113	0	2376	25
104 A	295	Glue	269	Glue	281	G+C																		
-													290	Glue	225	Glue	231	Glue	2679	25	2195	100	2380	83
104 B	280	Core	263	Core	277	Core							183	Glue	110	Glue	137	Glue	2660	100	2465	100	2330	100
104 C	300	Core	345	Face	271	C+F							190	Glue	160	Glue	186	Glue	2550	85	2485	95	2335	95
104 D	280	Core	245	Core	260	Core							295	Core	275	Glue	284	G+C	2429	20	2370	40	2401	49
105	315	Face	300	Face	290	Face							285	G+C	209	G+C	230	G+C	2320	65	2050	80	2206	71
106 A	245	G+C	190	G+C	230	G+C							185	Glue	190	Glue	161	Glue	2439	100	2260	100	2371	99
106 B	275	G+C	196	Core	265	Core							275	Glue	200	Glue	240	Glue	2829	5	2590	5	2724	92
107 A	300	Core	280	Core	292	Core							169	Glue	140	Glue	158	Glue	2793	100	2450	100	2605	68
107 B	360	Core	280	Core	330	Core							280	Glue	260	Glue	270	Glue	2700	100	2190	100	2370	70
107 C	325	Core	285	Core	306	Core	203	Glue	185	Core	183	C+G	260	Glue	150	Glue	208	Glue	2798	100	2123	29	2430	80
107 D	333	G+C	290	G+C	333	G+C	160	Glue	160	Glue	160	Glue	290	Core	230	Glue	275	Glue	[illegible]	100	1870	.70	2041	40
108 A	335	Core	310	Face	302	C+F	170	Glue	168	Glue	168	Glue	200	Glue	180	Glue	190	Glue	2240	95	2019	98	2183	94
108 B	310	Core	270	Face	287	C+F							298	Core	282	Core	288	G+C	2330	100	2290	100	2396	100
109 A	370	Face	315	Face	347	Face	135	Glue	100	Glue	130	Glue	289	Glue	260	Glue	271	Glue	2330	5	1763	43	2037	50
109 B	326	Core	275	G+F	298	C+F	190	Glue	150	Core	163	G+C	222	Glue	160	Glue	193	Glue	2140	10	1539	50	1891	40
110 A	330	Core	285	Core	300	Core	160	Glue	143	Glue	156	Glue	273	G+C	209	Glue	246	Glue	2320	36	1555	55	2008	70
110 B	335	Core	300	Face	319	C+F	170	Glue	160	Glue	166	Glue	219	Glue	195	Glue	203	Glue	2400	35	2180	90	2272	73
111 A	319	G+C	267	G+C	309	G+C	195	Glue	163	Glue	179	Glue	280	Glue	270	Glue	275	Glue	2340	60	2360	15	2442	30
111 B	379	Face	270	Face	296	Face	185	Glue	150	Glue	179	Glue	200	Glue	180	Glue	190	Glue	2215	40	2039	85	2131	41
112	320	Face	190	Face	172	Face	160	Glue	150	Glue	155	Glue	195	Glue	180	Glue	188	Glue	2700	95	2212	90	2400	92
113 A	320	Core	300	Core	306	Core	180	Glue	160	Glue	160	Glue							3087	25	2090	90	2394	70
113 B	295	G+C	250	G+C	271	G+C	195	G+C	170	G+C	178	G+C							2475	75	2312	15	2386	97
113 C	330	Core	260	Core	292	Core					115	Glue							2875	15	2662	10	2771	18
113 D	240	Glue	180	Glue	210	Glue	175	Glue	190	Glue	163	Glue							2687	70	2087	60	2330	60
114	319	Core	275	Core	292	Core	130	Glue	140	Glue	145	Glue	325	Glue	300	G+F	308	G+F	2812	65	2337	50	2662	60
115 A	225	Face	178	Face	191	Face	170	Glue	155	Glue	162	Glue	190	Glue	140	Glue	166	Glue	2725	100	2325	10	2620	62
115 B	325	Glue	275	Glue	276	Glue	185	G+C	170	Glue	106	Glue	225	Core	205	Core	212	Core	2625	100	2275	98	2469	81
116	340	Core	305	Core	320	Core	200	G+C	175	Glue	190	Glue							2337	100	2337	100	2362	100
117	315	Face	244	Face	281	Face	200	Glue	165	Glue	176	Glue	209	Glue	140	Glue	181	Glue	2890	100	1490	100	2668	70
118	279	Face	180	Face	201	Face	233	Glue	188	Glue	218	Glue	230	G+C	180	G+C	210	G+C	2650	35	2323	5	2466	43
119	320	Face	235	Face	277	Face	233	G+C	225	G+C	231	G+C	180	Glue	170	Glue	178	Glue	2525	90	1988	90	2217	89
121 A	300	Core	270	Core	240	Core	203	Glue	180	Glue	170	Glue	133	Glue	120	Glue	170	Glue	2330	100	2180	100	2271	72
121 B	310	Core	280	Core	292	Core	220	G+C	170	G+C	200	G+C	230	G+C	140	G+C	208	G+C	2487	95	2225	40	2330	74
122 A	260	Face	218	Face	232	Face	200	Glue	190	Glue	197	Glue	298	Glue	230	G+C	251	G+C	2725	10	2212	30	2426	32
122 E	330	Face	200	Face	279	Face	305	G+C	260	Glue	290	Glue	[illegible]	Glue	[illegible]	Glue	[illegible]	Glue	2700	95	1800	10	2087	32
123 A	370	Core	310	Core	338	Core	220	Glue	150	Glue	[illegible]	Glue	190	Glue	170	Glue	180	Glue	2375	95	2187	90	2437	81
123 C																			2787	5	2097	0	2509	8
123 E	210	Face	200	Face	205	Face	240	Glue	215	Glue	[illegible]	Glue	[illegible]	G+C	210	Glue	267	Glue	3150	100	2837	100	2984	90
124 A	390	Core	300	Core	326	Core	200	Glue	185	Glue	191	Glue	240	G+C	[illegible]	G+C	228	G+C	2712	100	2300	100	2531	100
124 B	375	Core	320	Core	340	Core	275	G+C	250	G+C	266	G+C	240	G+C	145	Glue	213	G+C	2612	100	1800	60	2215	82
125 A	265	Face	250	Face	256	Face	175	Glue	160	Glue	[illegible]	Glue	120	G+C	190	Glue	230	G+C	2625	70	1730	40	2008	86
125 B	309	Core	240	Core	283	Core	260	G+C	140	Glue	213	Glue	183	Glue	[illegible]	Glue	[illegible]	Glue	[illegible]	[illegible]	2075	100	2314	64
126 A	266	Face	240	Face	255	Face	205	Glue	130	Glue	176	Glue	250	G+C	190	G+C	219	G+C	2537	10	2262	95	2332	74
126 B	360	Core	315	Core	333	Core	180	Glue	140	Glue	[illegible]	Glue	205	G+C	180	G+C	[illegible]	G+C	2729	100	2300	95	2382	76
126 C	333	Glue	290	Glue	322	Glue	200	Glue	[illegible]	Glue	200	Glue	230	G+C	240	G+C	242	G+C	[illegible]	5	2300	100	2512	31
126 D							185	Glue	100	Glue	168	Glue	277	G+C	120	G+C	219	G+C	2750	100	2337	90	2683	70
127	310	Core	280	Core	297	Core	220	Glue	200	Glue	210	Glue	255	G+C	180	G+C	211	G+C	2862	10	2475	11	2609	33
128 A	318	Face	215	Face	270	Face	210	Glue	185	Glue	211	Glue	255	G+C	220	G+C	227	G+C	2862	100	2300	90	2350	70
31																			2390	100	1895	20	2108	48
32																			2350	50	1855	0	2037	8
33																			2300	50	1910	30	2156	39
34																			2210	10	1910	10	1906	43

*

Ⓐ Indicates no separation of the plies

Ⓑ Indicates that the total separation between all the plies, regardless of the number of plies, is not greater than ten per cent of the area of sample tested.

Ⓒ Indicates a total separation greater than ten per cent and not more than fifty per cent of the area of the sample tested or indicates blisters or other manufacturing defects of such nature as would warrant further tests.

Ⓓ Indicates a total separation of over fifty per cent of the area of the sample tested

LEGEND

(‡) Includes five miscellaneous (XX-31-32-33-34) Caseins not prepared by "Dahlberg"

(b) Washed four times, ground and rewashed four times

\# Calculated as Lactic Acid